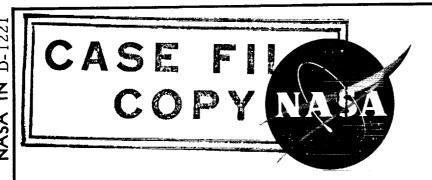
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TECHNICAL NOTE

D-1221

INFLUENCE OF CERTAIN COMPOSITION AND FABRICATION

VARIABLES ON THE STRESS-RUPTURE PROPERTIES

OF A COBALT-BASE ALLOY CONSOLIDATED

BY POWDER METALLURGY

By Philip A. Clarkin, John W. Weeton, and Paul F. Sikora

Lewis Research Center Cleveland, Ohio

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SUMMARY

An investigation was conducted to determine the effect of carbon content and type of carbide formation and distribution on the stress-rupture properties and on the forgeability of an S-816 powder-metallurgy alloy. Liquid-state-sintering techniques were used as well as heat treatments and mechanical working in order to control the carbide formations and distributions.

The majority of specimens produced had lives longer than that of standard wrought S-816 tested under the same conditions (25,000 psi at 1500°F). This was true for specimens with higher and lower carbon contents than 0.4 percent, the normal percent utilized in the wrought product. Highly forgeable structures were produced; even specimens containing as much as 1 percent carbon were successfully hot-swaged to 50 percent reduction in area. Stress-rupture properties generally increased with carbon content, but simultaneously were influenced by conditions of work and heat treatment. The microstructures for best stress-rupture strength depended on the range of carbon content.

¹A condensed version of this report, entitled "Effects of Variations in Carbon Content, Heat Treatment, and Mechanical Working on Stress-Rupture Properties of a Liquid-Phase-Sintered, High-Temperature Alloy," was presented at the Fall Meeting of the Metallurgical Society of the American Institute of Mining, Metallurgical, and Petroleum Engineers, Inc., Philadelphia, Oct. 17-20, 1960, and was published in the February, 1962, issue of the Transactions of the Society, vol. 224, pp. 116-120.

INTRODUCTION

Most of the commercial advantages and disadvantages of powder metallurgy have been discussed many times by others. However, from a research standpoint, powder-metallurgy methods may be considered a means of obtaining structures that are not obtainable using normal ingot and mechanical working. Structures normal in cast alloys may sometimes be avoided when the alloys are made from powder. For example, it has been shown (ref. 1) that massive grain boundary carbide networks, common to cast X-40, may be avoided by making the alloy from prealloyed powders. Such carbide networks in cast structures tend to make the alloy brittle and in many cases definitely prevent the alloy from being forged. Thus, alloys that cannot readily be forged when produced by conventional practice may possibly be forged when the alloys are made by powder-metallurgy techniques.

Another problem that is quite similar to the "brittle" network problem is that cast materials, particularly ingots, are subject to segregation of both brittle components and alloying elements in dendritic interstices; such segregations can be especially troublesome and harmful during heat treatment. For example, it has been shown that, because of differences in segregation, the response to heat treatment of an alloy casting of one size and shape may be completely different from the response of the same alloy cast in a different size or shape. An instance of this occurred with cast HS-21 alloy (refs. 2 and 3). It was found that a heat treatment that improved the life of small cast HS-21 alloy turbosupercharger buckets seriously damaged larger turbojet buckets of the same alloy. The temperature of the heat treatment was high enough to cause grain boundary melting in the large buckets but did not have this effect on the smaller buckets.

These differences in response to the same thermal treatment were attributed to differences in carbide segregations between the small and large castings. Again, by utilizing powder-metallurgy techniques, it may be possible to avoid such differences in segregations by artificially dispersing the carbide-forming constituents uniformly throughout the microstructure of the material.

Segregation also results in the tying up of valuable alloying elements. For example, in some alloys massive carbides that form during ingot practice may never be fully put into solution, and, therefore, their alloying elements are not available for age hardening of the alloys (ref. 4). A typical alloy where several percentages of alloying elements may be tied up by carbide segregations that occurred in the ingot processing is the wrought alloy S-816. Approximately half the strengthening elements niobium and tantalum, as well as some chromium, molybdenum, and tungsten are tied up in inert form throughout the entire thermal history of the alloy (ref. 5). Alloy S-816 has been used for many years as a turbojet bucket alloy. It has a high degree of versatility, in that it responds well to solution-strengthening, hot- or cold-working, and precipitation-strengthening. In preliminary experiments, an alloy with a composition identical to that of S-816 except that it had no carbon

was cold-worked and tested at 1500° F (unpublished NASA data). It showed considerable strength relative to conventionally produced S-816 (nominally 0.4 percent carbon).

Because of the potential benefits that might be derived by use of powder-metallurgy methods, it was decided to combine prealloyed powder of a high-temperature alloy with varying quantities of powdered carbon. An alloy powder was selected that had the composition of wrought S-816 minus carbon. Varying quantities of powdered carbon were admixed, and the resulting microstructures were studied in relation to their stress-rupture properties.

Specifically, the objectives of this investigation have been to determine:

- (1) The effect of varying carbon content on the stress-rupture life and ductility of S-816 type alloys made from powders
- (2) The effect of different carbide formations, resulting from variations of carbon content, heat treatments, and mechanical working treatments, on the stress-rupture and ductility of a series of S-816 type alloys made by powder-metallurgy methods
- (3) The effect of varying carbon content on the workability of the S-816 type alloy produced by powder-metallurgy methods
- (4) Any improvement in properties that could be obtained by dispersing alloying elements and precipitates by powder-metallurgy methods rather than by conventional fabrication methods

For the present investigation, nearly carbon free S-816 alloy was cast and subsequently remelted and powdered. The compositions of powdermetallurgy compacts were varied by adding graphite to the basic material in different percentages. Carbon content was varied from 0.1 to 1.0 percent. In order to produce carbide formations and dispersions different from those produced by sintering alone, specimens were heat-treated, hotswaged, or hot-swaged and heat-treated. Data were obtained on the stress-rupture lives and ductilities of these specimens, and metallographic studies of their microstructure were made.

MATERIALS, APPARATUS, AND PROCEDURE

Raw Materials

The master alloy powder was made from elements cast into low-carbon S-816-type-alloy ingots, which were remelted and powdered by a commercial

concern. (A small residual carbon content was left in the structure because the cobalt initially contained some carbon.)

About 60 pounds of alloy in the form of ingots 1 by 1 by 3 inches were powdered. The powdering process consisted of melting the ingots under argon in a zircon crucible, tapping the bottom to produce a thin stream of molten alloy, and atomizing the stream by means of a high-pressure water spray. The powder was passed through a collecting, dewatering, and sieving system to yield particles of 100 mesh or finer. From the 60 pounds of alloy melted, about 15 pounds of less-than-100-mesh prealloyed powder were obtained. The chemical analysis of the powder is listed in the following table in percentages along with the nominal composition of standard S-816:

	Cr	Ni	C b	Мо	W	Fe	Co	C
Alloy powder used in this in- vestigation	18.88	19.56	4.53	4.24	2.90	2.64	45.39	< 0.01
Standard S-816	20.0	20.0	4.0	4.0	4.0	3 . 0	43.0	0.4

High-purity graphite was mixed with the alloy powder to obtain specimens with various carbon contents.

Cleaning of Powders

A sample of the as-received powder was compacted, sintered, and examined metallographically for oxide content. Although the surfaces of the as-received powders had a silvery appearance, the sintered specimens showed that a fair amount of oxide remained on the surfaces. Since this type of oxide film could be damaging to the ultimate properties of the material, it was removed by reduction with hydrogen (-100° F dew point). The apparatus used for the reduction process was the rotating tube furnace described in reference 6. In this apparatus the reduction took about 8 hours per pound at 2100° F. For the present investigation, about 10 pounds of powder were cleaned by this method.

Fabrication of Specimens

To make a single specimen, 100 grams of powder were mixed with 0.5 gram of paraffin binder dissolved in benzene; to this a predetermined amount of graphite was added. The slurry was then heated and stirred in a casserole until the benzene was completely evaporated. This method of

mixing graphite and alloy powders produced more homogeneous mixtures than dry-mixing and ball-milling methods. By adding various quantities of graphite, specimens containing nominally 0.1, 0.2, 0.3, 0.4, 0.5, 0.75, and 1.0 weight percent carbon were made. After the powder-graphite mixture was completely dry, it was sealed in rubber tubing and hydrostatically pressed at 25 tons per square inch. The pressed compact that resulted was about 3 inches long and 5/16 inch in diameter. For sintering, the pressed compact was placed in an alumina sleeve that was in turn inserted vertically into a graphite block. The entire assembly was then heated by induction in a vacuum of about 10 to 20 microns of mercury. temperature of the specimen was slowly raised while the specimen was viewed through an observation port in the top of the furnace. Close observations were made to determine the point at which the compact would just begin to melt; this point could be detected by noting a change in the reflections of the top surfaces of the specimens. After incipient melting was first observed, the temperature was held constant for approximately 5 seconds and then lowered. By sintering in this manner, melting occurred only at the surfaces of the individual particles in the compact. The interiors of the particles remained solid during the entire sintering process. This quasi-liquid-phase sintering generally resulted in dense specimens that retained their initial pressed shape. In some instances, however, metal or paraffin vapors fogged the observation port, and the melting point could not be clearly determined. When this happened, incipient melting could not be readily detected, and the specimens either were not densified sufficiently or melted completely.

Stress-Rupture Tests

Test specimens were machined from the alloys to the dimensions shown in figure 1 and then checked for surface defects by zyglo techniques. All specimens were tested in stress-rupture at 1500° F at a stress of 25,000 pounds per square inch.

Specimen density was determined before stress-rupture testing, and the microstructure of the specimens was examined before and after testing. After the stress-rupture tests, part of each broken specimen was cleaned of surface oxide and chipped for carbon analysis.

Fabrication Treatments Given Specimens

The specimens were tested in the following conditions:

- (1) As-sintered
- (2) Heat-treated (solution-treated 24 hr at 2150° F, water-quenched; aged 16 hr at 1400° F, air-cooled)

(4) Hot-swaged from 2150° F (reduction in area of about 50 percent) and heat-treated (solution-treated 24 hr at 2150° F, water-quenched; aged 16 hr at 1400° F, air-cooled)

Actually, the hot-swaging consisted of heating the specimens at 2150° F and placing them in the swaging machine. After one or two blows, the temperature of the specimen had dropped considerably, and at this point the specimen was reheated. Therefore, although attempts were made to keep the temperature of the specimen as close as possible to 2150° F, some swaging was done at a temperature lower than 2150° F. Several reheatings were necessary to swage the specimens completely.

RESULTS

Stress-Rupture Life as Function of Carbon Content

The stress-rupture properties of all specimens are listed in table I. The stress-rupture lives and ductilities of specimens are plotted as a function of carbon content in figures 2 to 5; data concerning some specimens that broke on loading are not included in these figures. As a point of reference, the average stress-rupture life of wrought S-816 (given treatments of AMS 5765A) is indicated in each figure. The average value, about 70 hours under the test conditions used in this investigation, is representative of numerous tests reported in the literature. It is known that S-816 bar stock and forged products made from S-816 are sensitive to both thermal and mechanical variables sometimes introduced during commercial fabrication practices and that scatter in stress-rupture life values for the alloy may on occasion be large. It is thus as difficult to determine a representative scatter band for the lives of the wrought product as it is to determine an exact average for the alloy. Since the average life of the wrought S-816 for the test conditions of this investigation (25,000 psi at 1500° F) is to be used as a basis for comparison throughout the report, reference 7 was selected from available literature to represent the properties of the wrought product. Comparisons of lives of powder-metallurgy specimens with the "standard" are made only to indicate the adequacy or superiority of the powder products (where they exist) to the selected "average standard" value. In the figures representing stress-rupture lives, arrows are drawn from the point representing the standard value to further indicate the uncertainty of the value.

As-sintered specimens. - Figure 2 shows stress-rupture properties of as-sintered specimens as a function of carbon content. The large amount of scatter in stress-rupture life can be attributed to several factors. First, scatter in properties is inherent in the stress-rupture test itself. Second, the fabrication methods used to make these specimens often led to microstructural differences in specimens that were intended to be identical. Third, density variations undoubtedly contributed to scatter;

however, data obtained in this investigation did not allow an unequivocal correlation between differences in stress-rupture properties and differences in densities.

Because of the amount of scatter, stress-rupture curves are drawn through both upper and lower data points and a scatter band of life values is presented. The life values at the lower edge of the band are quite good with respect to the average life of wrought S-816, indicated by the solid point; values at the upper edge of the band indicate the high potential of the powder-metallurgy process. The stress-rupture life tends to increase with increasing carbon content, the very low carbon specimens having lives roughly equivalent to the average life of the wrought alloy under these test conditions.

Heat-treated specimens. - Figure 3 shows the stress-rupture lives of specimens that had been heat-treated after sintering. For the specimens with lower carbon content, the scatter in properties was much less than that for specimens with higher carbon content. Almost all specimens with carbon contents greater than 0.4 percent had stress-rupture lives above that of wrought S-816. The curve shows a general upward trend of strength with increasing carbon content. For specimens containing approximately 0.56 percent carbon, there was a wide range of stress-rupture life that extended from approximately 50 to about 210 hours. At carbon contents of approximately 0.80 to about 0.84, the scatter range extended from 100 to 652 hours. The specimen that ran for 652 hours was the best tested in this investigation.

Hot-swaged specimens. - Figure 4 shows the results of stress-rupture tests of specimens that were hot-swaged after sintering. The scatter in properties of these specimens is less than the scatter for the as-sintered or heat-treated groups. This is not surprising since one would expect swaging to reduce the porosity that is present after the sintering process. There is again an increase in stress-rupture life as the carbon content of the material is increased. In this group of specimens, only those with carbon contents of more than 0.4 percent had stress-rupture lives significantly longer than that of the wrought product.

Hot-swaged and heat-treated specimens. - Figure 5 shows the results of stress-rupture testing of specimens that were hot-swaged and heat-treated after being sintered. Stress-rupture life again increases with increasing carbon content, and, as in the case of the material that had been heat-treated only, there is wide scatter in the data for certain carbon percentages. One specimen had an unusually long life (145 hr) for its carbon content of 0.08 percent. Another extremely strong specimen, with a carbon content of 0.65 percent, had a life of approximately 625 hours. At this same carbon content another specimen had a life of only 240 hours. Later in this report reasons are given that may account for the differences in properties between these high- and low-strength

specimens at given carbon contents. In this group of hot-swaged and heat-treated specimens there were two out of eight with carbon contents less than 0.4 percent that had stress-rupture lives equal to or better than that of the wrought product. All the specimens with carbon contents above 0.4 percent had better lives than the wrought specimen.

Ductility of Stress-Rupture Specimens

Curves showing ductility (reduction in area) as a function of carbon content for each group of stress-rupture specimens have been plotted in the same figures as the respective life curves (figs. 2 to 5), and ductility of each specimen is also shown in table I. An accurate comparison of these ductility values with that for the wrought alloy under the same test conditions is difficult because of the large amount of scatter in reported ductility and the lack of a well established scatter band. A rough comparison may be based on a ductility value for the wrought alloy of about 10 percent reduction in area.

As-sintered specimens. - In general, as shown in figure 2, as the carbon content increases, stress-rupture life of the as-sintered specimens increases, and the ductility (percent reduction in area) decreases. It is interesting that, for the higher carbon content specimens, for which stress-rupture lives are very widely scattered, the ductility values are very similar; that is, two specimens having the same carbon content may have widely differing lives yet the same ductility values.

Heat-treated specimens. - The ductility curve for heat-treated specimens shows a maximum at 0.2 percent carbon. For carbon contents above 0.2 percent, there is a decrease in ductility with an increase in both carbon content and stress-rupture life. Note again that at higher carbon contents, where there are widely divergent stress-rupture lives, the ductility curve shows no differences relating to the rupture lives.

Hot-swaged specimens. - The ductility of swaged specimens as a function of carbon content is shown in figure 4. As in the cases of the assintered and heat-treated groups of specimens, the swaged group exhibits a peak in its ductility curve. This peak occurs at about 0.3 percent carbon. As can be seen in figure 4, the swaged specimens had a large amount of scatter in ductility values.

Hot-swaged and heat-treated specimens. - Since there was little scatter in stress-rupture lives of hot-swaged and heat-treated specimens at a given carbon content (fig. 5), it is not too surprising that the ductility values also fall rather closely about a single curve. One specimen having a very low carbon content, less than 0.1 percent, had a very long stress-rupture life and low ductility. In specimens having a high carbon content, approximately 0.65 percent carbon, three specimens exhibited rupture lives

ranging from approximately 230 to 620 hours. Among these specimens, those that had short stress-rupture lives had high ductilities, and the specimen that had a very long rupture life of 620 hours had the lowest ductility.

Density of Specimens

The densities of specimens are listed in table I. For comparative purposes, the density of each specimen is given as a percentage of the wrought-alloy density ($8.59~\rm g/cm^2$). Fifty-four of the specimens had densities greater than 90 percent of the wrought-alloy density, and most of these had almost the same density as that of the wrought alloy. Six specimens had densities between 80 and 90 percent of the wrought-alloy density, and two had densities below 80 percent. One of the latter specimens was in the as-sintered group, while the other was in the heat-treated group. No specimens that were worked by swaging had densities below 80 percent of the wrought-alloy density.

The average density of the specimens in each group (percent of wrought-alloy density) was as follows: as-sintered, 94.4 percent; heat-treated, 96.6 percent; hot-swaged, 97.9 percent; hot-swaged and heat-treated, 98.8 percent.

Workability of Powder-Metallurgy Specimens

The workability of powder-metallurgy specimens produced in this investigation was very good. While no systematic studies of workability were made, it was found that a 50-percent reduction in area by hot-swaging was easily accomplished. Even specimens containing as much as 1.0 percent carbon were successfully hot-swaged.

Studies of Microstructures

Studies were made of the microstructures of all specimens in the untested or as-fabricated conditions and of the structures of the test specimens after the tests had been run. Photomicrographs of almost every specimen for which data are plotted in figures 2 to 5 are shown in figures 6 to 9.

The photomicrographs of figures 6 to 9 were made at the relatively low magnification of 250. At this magnification it was possible to see overall carbide structures, voids, fracture edges in the test specimens, and even some precipitation in the matrix of the grains. In addition to the low-magnification photomicrographs, others (figs. 10 to 13) were made

at a magnification of 750 to show details of the structures of some selected specimens. Most of the latter photomicrographs were of specimens that had significant differences in physical properties that appeared to correlate, to some degree, with microstructures.

It should be pointed out that during the initial sintering treatment a temperature gradient along the specimen was unavoidable; hence, some parts of the specimen undoubtedly experienced greater liquation than others. Therefore, it is not surprising that microstructural differences may be apparent between some of the photomicrographs of as-sintered specimens, which were taken at sections cut from tops of specimens, and the photomicrographs of test specimens, which were taken at sections cut from the centers of specimens. On a gross scale, such differences did not affect conclusions drawn from studies of the microstructures.

As-sintered specimens. - The microstructures of as-sintered specimens are shown in figure 6. Photomicrographs show that much of the carbon in each specimen is in grain boundaries in the form of spherical carbides, massive carbides, Chinese script, or eutectic areas. Massive formations of carbides are evident in grain boundaries of the higher carbon specimens, such as specimens 13 to 17 (figs. 6(k) to (o)). Also, high-carbon specimens contained more spheroidized carbides within the grains than did lower carbon specimens (specimens 1 to 4).

It may also be noted that there is considerable microporosity in some of the as-sintered specimens. Some specimens, such as specimens 11, 13, and 14, exhibit porosity in photomicrographs of both the as-sintered and the tested structures. In other specimens, such as specimens 15 and 16, porosity is evident only in the photomicrographs taken of the specimens after testing. In specimen 17 it is interesting that the porosity near the failed section of the test specimen was considerable, whereas there were few holes in the portion of the specimen that had not been tested. As is discussed later, the porosity of the area under stress is obviously more important than the porosity in a section that is not stressed. The porosity of the test section, of course, could not be determined from metallographic examination of specimens before testing.

Some of the specimens were inadequately sintered. For example, specimens 5 to 7 fell into this category and had unusually large voids. Only specimen 5 is shown, since the others are almost identical. It is mentioned earlier that tests were run on all these specimens and that the data are listed in table I. Where porosity was very great, as in specimens 5 to 7, the specimens broke on loading; these results were not entered in plots of stress-rupture life against carbon content.

There was definite evidence of eutectic formations in boundaries of some specimens. That is, eutectic structures were formed that contained fine lamellae (see specimens 11 and 16). Such formations usually respond

to high-temperature heat treatments more readily than do the more solid or massive carbides.

Heat-treated specimens. - Figure 7 shows the microstructures of specimens in the heat-treated condition before and after testing. solution treatment given the specimens (24 hr at 2150° F) partially dissolved some of the massive carbide formations. In particular, it completely eliminated all finely laminated structures, partly dissolved massive carbide formations in boundaries, and produced spheroidized carbides in grain boundaries and within grains. It was surprising that some Chinese script formations were not eliminated (see specimen 20, fig. 7(b)) because other studies (ref. 4) showed that they were rather easily dissolved. In low-carbon specimens almost all carbides were distributed in the grain boundaries rather than within the grains, whereas in higher carbon specimens residual carbides often existed in the interiors of the grains as well as in the grain boundaries. (Residual carbides refer to those present in the structure before stress-rupture testing and not carbides that are present as a result of precipitation during the stressrupture test.) In general, the as-sintered microstructures of highcarbon specimens were changed more drastically by the heat treatment than were the as-sintered microstructures of low-carbon specimens. be seen by comparing the microstructures of high- and low-carbon specimens of the heat-treated group of figure 7 and the as-sintered specimens of figure 6.

Hot-swaged specimens. - Figure 8 shows the microstructures of hotswaged specimens. For the most part, hot-swaging has broken up massive formations of carbides or the eutectic area that was present in the assintered condition. The hot-swaging also refined the grain size. virtue of the fact that the larger carbide formations were broken up, the hot-swaging also improved the gross uniformity of the carbide distribution throughout the structure. The type of carbide being referred to here is for the most part a residual carbide rather than a carbide that precipitated upon aging. Perhaps the most significant contribution of the swaging operation was that it eliminated a good percentage of the microporosity. Some of the specimens, such as specimens 37 and 43 in figures 8(d) and (j), appeared to be fairly well solution-treated in the as-swaged condition. This is not too surprising, since the swaging operation was carried out at a temperature of 2150° F, and since the swaging necessitated multiple reheatings of the specimens into the solutiontreating temperature range. Again, Chinese script structures were evident in low-carbon specimens (specimens 35, 36, 38, and 39).

Hot-swaged and heat-treated specimens. - The microstructures of specimens that were hot-swaged and heat-treated are shown in figure 9. The combination of the two types of treatments, in most cases, almost completely eliminated large carbide and eutectic areas in the tested areas of specimens. A few areas where the carbides are segregated in grain

boundaries are, however, evident in specimens before the tests. In general, the residual carbides in the specimens are small, and in the lower carbon specimens are located at the grain boundaries. In high-carbon specimens carbides are present within the grains as well as at the grain boundaries. There is a negligible amount of microporosity in all specimens in the heat-treated and hot-swaged group. Many of the specimens in this group have microstructures that closely resemble that of standard wrought S-816. The microstructure of specimen 57 particularly resembles that of wrought S-816.

DISCUSSION

Significance of Stress-Rupture Properties

It is evident from the stress-rupture test results that the powdermetallurgy processes used in this investigation produced many specimens with significantly greater stress-rupture lives than the average stressrupture life of wrought S-816. A value of 70 hours was felt to represent the life of standard S-816, given the fabrication and heat treatment of AMS 5765A, although it is recognized that the wrought commercially made S-816 may exhibit a large amount of scatter of stress-rupture lives and that the upper values for such materials could well be above the average indicated here. It should also be pointed out that the stress-rupture life of wrought S-816 may be significantly improved by heat treatments. For example, some early work done on the effects of different solution treatments upon the stress-rupture properties of S-816 (ref. 8) showed that, under test conditions comparable to those used in the present study, lives ranged from approximately 100 to 200 hours in specimens solutiontreated at temperatures of 2300° and 2350° F. This early work also contains some properties of cast S-816; values of stress-rupture life were widely scattered, ranging from approximately 200 to over 1500 hours.

Stress-rupture lives of wrought, wrought and heat-treated, and cast alloy specimens may thus be greater than the "standard" value used as a basis for comparison. It is believed that the lives of powder-metallurgy specimens are for the most part very good, particularly since the specimens were made by a laboratory practice and since it was not within the scope of this paper to optimize properties by varying sintering temperatures or conditions, heat treatments, or forging practices.

Using the carbon content of standard wrought S-816 (0.4 percent by weight) as a point of reference, it is interesting to note that most specimens that had carbon content equal to or greater than 0.4 percent had superior properties to those of the wrought material. It is also interesting to note that several of the specimens that had lower carbon contents than that of the wrought product had properties better than those of the standard product. These facts illustrate one of the potentials of

the powder-metallurgy process in that they show that an efficient use of the alloying elements in a given material may be made by means of powder metallurgy.

Forgeability of Powder-Metallurgy Specimens

It is well known that it is very frequently difficult to forge high-temperature alloys containing large quantities of carbon in combination with strong carbide forming elements. Usually castings or ingots made from such material form an embrittling network that prevents subsequent forging. It is of interest that materials containing large percentages of carbon, as much as 1 percent in some cases, made by the powder-metallurgy methods of this investigation could be hot-swaged. Since swaging practices may chill the material being swaged to a very low temperature rather quickly, it might be expected that other forging practices used with powder-metallurgy products might be even more easily accomplished. In any case, a valuable use of the powder-metallurgy process is that it is possible to avoid normal casting of ingots and concomitant macro or micro segregations that sometimes prevent forging.

Correlation of stress-rupture life with microstructure and degree of porosity. - It would be highly desirable to be able to predict the strength of alloys possessing a given microstructure. Correlations of microstructure with properties are, at best, difficult even when cast or wrought products are considered. One of the difficulties encountered in attempting to correlate microstructures of powder-metallurgy products with properties is that resulting from inherent porosity incurred during sintering.

Porosity in powder-metallurgy products presents a problem somewhat analagous to that of microporosity in cast alloys. In both cases gross density measurements are not always indicative of the strength or weakness of the material being tested. It is thus possible to have a test specimen having little overall porosity, but with concentrated porosity in the area of the specimen where it could be most harmful. Another factor associated with the correlations to be presented is liquation in the powders during sintering. This, of course, could produce anything from very subtle to gross differences in microstructure and thus affect properties. The melting point (liquation between particles) was detected by noting a change in color or luster of the surface of the powder product during sintering. Thermocouples on the specimens would not be expected to detect incipient melting in a powder product such as that studied. fact, since clean powders were made with difficulty in small quantities, and, since specimens varied greatly in their carbon contents and resultant melting points, it was not felt that elaborate attempts to detect

melting points accurately were warranted. It might be argued that the visual method used was an efficient way to detect incipient melting in relatively few specimens.

It should be realized also that not only would sintered specimens be affected by differences incurred as a result of variations of melting-point detection, but any powdered alloy of a given composition would be expected to respond somewhat differently to either heat treatments or forging (or both) if "as-sintered" conditions were different.

As the subsequent discussion unfolds, it is shown that certain consistent relations between microstructure and stress-rupture life exist, that porosity effects noted do not affect overall conclusions, and that different microstructural configurations evidently relating to stress-rupture results are probably largely initiated during the sintering process.

It was felt that some interesting observations could be made by comparing the microstructures of groups of as-sintered specimens of a given carbon content in which some specimens in the group had very low properties while the others had very high properties. The first comparison was made on three low-carbon specimens. Specimen 2 (containing 0.1 percent carbon) and a stress-rupture life of 89 hours, whereas specimens 1 and 3 had somewhat shorter stress-rupture lives of 43 and 51 hours, respectively. In this comparison, the differences in these stress-rupture lives are not too great and may well be within the normal scatter expected in the stress-rupture test. It was not too surprising, then, that a comparison of the microstructures of the specimens (fig. 10) revealed nothing to account for differences in stress-rupture lives. Also, there was no difference in density values that could account for the differences in lives. In fact, the best specimen had the lowest measured density.

Another pair of specimens that might be compared contained a higher carbon content. For example, the microstructure of a specimen containing 0.775 percent carbon (specimen 16, fig. 10(e)), which had a very high stress-rupture life of 485 hours, may be compared with the microstructure of a specimen with 0.80 percent carbon (specimen 13, fig. 10(d)), which had a relatively low stress-rupture life of 182 hours. Examination revealed that the longer lived specimen 16 had more massive grain boundary carbide formations than did specimen 13. After testing, the weaker specimen showed more aging and precipitation than did the stronger of the two specimens.

As-sintered specimens. - Prior to discussing the correlations of the as-sintered microstructures and properties of successfully tested specimens, it might be of interest to note that in those as-sintered specimens that broke upon loading (specimens 5, 6, and 7) the photomicrographs revealed that the powders were almost in an unsintered state, as shown in

figure 6(e) (specimen 5). It is important to notice how some of the carbon or carbides tend to segregate in areas where three grain boundaries meet. It is also interesting to note that many of the grain boundary areas contain only a fine interface of carbide or precipitates between two grains. The fact that many carbides are visible within the powder particles indicates that a very rapid diffusion of carbon into the alloy powder particles had taken place and that a degree of homogenization had probably taken place before the powders were welded or fused together. The densification of the powder product that occurs upon fusion, of course, is most desirable. However, if all the carbides were to form a continuous network surrounding the powder particles one would have difficulty in eliminating them by heat treatment. Furthermore, specimens with such a network would be difficult if not impossible to forge. It is evident throughout the discussion of the microstructures that no continuous networks of massive carbides were observed in the specimens regardless of their carbon contents. Densities of the different specimens did not vary significantly.

Heat-treated specimens. - Low-carbon and medium-carbon heat-treated specimens had rupture lives that were low relative to as-sintered specimens of comparable carbon contents. Visual examination of low-carbon specimens at a high magnification (750) reveals that many of the massive carbides that initially were present in the as-sintered state have been broken up, and many of them have been dissolved by the heat treatment. For example, structures of heat-treated specimens (specimens 18, 20, 21, and 23, figs. 11(a) to (d)) have fewer massive carbides in the boundaries than do as-sintered specimens (fig. 10). Also, the Chinese script structures have broken up to some degree.

Another interesting observation is that the low-carbon heat-treated specimens after tests show much more precipitation than do the low-carbon as-sintered specimens. Since the properties of the lower carbon heat-treated specimens are lower than those in the as-sintered specimens, it may be concluded that the grain boundary formations in the as-sintered specimens strengthen the alloy more than does solutioning and aging of these low-carbon specimens.

Any strengthening of the as-sintered specimens (during the stress-rupture test) by precipitation would be expected to be of a lesser magnitude than the strengthening due to precipitation within the heat-treated specimens during stress-rupture testing. This is understandable since the heat treatment alone should dissolve some of the grain boundary carbides, and subsequently, during testing, these could be expected to precipitate out. However, as was noted, the strengthening resulting from precipitation was undoubtedly not as great as the strengthening due to the inhibition of slip by the formations of carbides in the boundaries, which in the sintered specimens were more continuous than in the heat-treated specimens.

The high-carbon specimens that were examined before testing had relatively minor quantities of carbides in the boundaries, and they were more or less spheroidized. After testing, photomicrographs taken from areas close to the fractured edges showed the carbides within the grains were also spheroidized. Also, in the latter specimens (32 and 33) there were fairly good sized formations of carbides in the boundaries, as shown in figures ll(e) and (f). Specimen 33 had the best stress-rupture life obtained in this investigation. As such, it warrants study in comparison with others. Specimen 32 had very poor properties, but the same carbon content.

There is a difference between the microstructure of the best specimen (specimen 33) after testing and the poorer specimen (specimen 32) that possibly could account for differences in stress-rupture life. The best specimen, although it had fairly large carbides in the grain boundaries and also a fairly good number of residual carbides in the matrix of the grain, had fewer massive carbides in the boundaries than did the poorer of the two specimens, namely, specimen 32. This type of difference is believed to be significant and has consistency with observations made on other pairs of specimens in other groups, as will be shown subsequently. However, in this case the poorer of the two specimens, specimen 32, also had a very low density, less than 95 percent, whereas the better specimen had a density of over 100 percent. Density considerations, then, could account for the differences in the rupture lives of these two specimens, but the microstructural differences must be kept in mind.

A comparison of microstructures of good and poor specimens may be made for the lower carbon specimens represented in figure 7. Specimens 28 and 29, which contained about 0.56 percent carbon, are presented in figures 7(h) and (i). The poorer of the two, specimen 28, had more massive carbides in the grain boundaries of the portion of the specimen that had been stress-rupture tested, whereas the better of the two, specimen 29, which had approximately three times the life of specimen 28, had fewer massive carbides in the grain boundaries. Although the poorer specimen had a slightly lower density than the better one (97 compared with 100 percent), the microstructure in this case is believed to be the more significant factor in explaining the differences in the properties of the two specimens.

Hot-swaged specimens. - The rupture lives of several swaged specimens were superior to those of the heat-treated specimens in the low-carbon and intermediate-carbon ranges. The microstructures of some of the lower carbon swaged specimens (before tests) shown in the high-magnification photomicrographs of figures 12(a) to (d) (specimens 34, 35, 38, and 39) were similar to the microstructures of the as-sintered specimens; that is, there were Chinese script structures and massive carbide formations. This similarity was somewhat surprising, since one would expect these

formations to be broken up by the swaging. Observations of specimens after testing showed that there was a considerable breaking up of carbides and a banding of the structures. Even though the swaging was conducted at a solution-treating temperature of 2150° F, the alloy did not age during the test as the heat-treated specimens did. For example, compare specimens 34, 35, 38, and 39 (figs. 12(a) to (d)) with specimens 18, 20, and 21 (figs. 11(a) to (c)).

The fact that the swaging operations, which were conducted at a temperature of 2150° F, did not eliminate the as-sintered structure completely indicates that the total solution time at 2150° F during the multiple heating and swaging treatments was not very great. As will be shown later, none of the as-sintered structures remained in low-carbon specimens that were heat-treated at 2150° F for 24 hours after swaging. Thus, the heat treatments incidental to swaging, at least for the low-carbon specimens, did not in themselves significantly alter the microstructures. In the case of the higher carbon specimens, which were swaged in the same manner as low-carbon specimens, the heat treatments involved in the swaging operation seemed to have a much greater effect on the structure; no longer did as-sintered formations prevail as they did in low-carbon swaged specimens. In view of the fact that the hot-swaging temperature was closer to the melting points of the high-carbon specimens than to those of the low-carbon specimens, these observations can be rationalized.

Two swaged specimens that might be compared are those containing 0.18 and 0.185 percent carbon, specimens 38 and 39, respectively. The photomicrographs of these specimens taken before testing are quite similar (figs. 12(c) and (d)), and any differences in properties could not be attributed to microstructural differences seen in these photomicrographs. The photomicrographs of the fractured specimens show, however, that the better specimen, the one that failed in 60.7 hours, had relatively few carbides distributed either in the grain boundaries or within the grains. The poorer of the two, which failed in 38.8 hours, had more massive carbides in the grain boundaries and a few more carbides in other areas of the test section. Although the differences were not gross, again there is a consistent pattern between these specimens and others previously described; that is, the weaker of the two specimens had larger carbide particles. It should be noted, also, that the densities of the two specimens being compared were almost identical.

A more interesting comparison may be made for specimens containing approximately 0.73 percent carbon. Referring to figure 8(k), with relatively low magnification photomicrographs, it may be seen that specimen 44, which fractured in 208.2 hours, had fewer grain boundary carbides both before and after the test than did specimen 45, which had a poorer life of 126 hours. Again, the picture is consistent with what has been observed in heat-treated or swaged groups previously; that is, of a pair of specimens that have differences in rupture life, the better specimen

has fewer massive carbide formations than does the poorer. The overall density measurements again show an anomaly. The poorer of the two specimens has a high density of 103 percent, and the better of the two a very low density of 92 percent. A difference in density does not show up in the photomicrographs of test sections of the specimens, and thus, the microstructure must be considered as possibly more significant than the density values in this case.

Hot-swaged and heat-treated specimens. - The curve of stress-rupture life as a function of carbon content rises more rapidly for the hot-swaged and heat-treated specimens than for either the swaged or heat-treated groups of specimens.

The most significant observation about the microstructures of almost all of the specimens in this group is that most of the structures formed during the sintering process have been broken up or changed considerably by the combination of hot-swaging and heat treatment. The remaining carbides present in the swaged and heat-treated specimens are, generally, residual ones that exist at grain boundaries and, in some cases, within grains. Most of the carbides are spheroidized or partially spheroidized, and no vestiges of massive or partial network structures remain. Since the specimens of this group, particularly the high-carbon ones, have rather long rupture lives, it may be concluded that this alloy base composition can obtain considerable strength from something other than massive formations of carbides in boundaries.

A low-carbon specimen with 0.08 percent carbon had a very long life of $143\frac{1}{2}$ hours. A few residual carbides were present in the specimen before the test in the boundaries in relatively random spots. During the test, carbides precipitated throughout the grains, on slip planes, and at grain boundaries. These structures are shown in figure 13(b) (specimen 49). Another very long lived low-carbon specimen (0.1 percent) is shown in figure 13(c) (specimen 50). The photomicrograph of the tested specimen shows some precipitation inside the grains and at grain boundaries. Shorter rupture lives were obtained in several of the slightly higher carbon specimens; for example, figures 13(a), (d), and (e) (specimens 48, 52, and 53) show the structures of such specimens. The only structural differences that were seen which could explain property differences between the good specimens (49 and 50) and the poor ones (52 and 53) were the rather extensive grain boundary precipitates evident in specimens 49 and 50 which were absent in specimens 52 and 53.

The very low life of specimen 48, which contained only 0.015 percent carbon, may possibly be attributed to its low density of 95 percent rather than to its microstructure. The densities of the two best specimens were 100 percent, and the density of the two poorer specimens were 89 and 101 percent. These densities may have some effect on the low life of the

specimens, although there is no consistent correlation of rupture life with density in this group.

As the carbon contents of this series of specimens are increased, more and more residual carbides are apparent at high magnifications (×750, fig. 13). Some matrix precipitation, although it is very faint, is evident in test sections of specimens having carbon contents greater than 0.6 percent. Again, in the specimens with a nominal carbon content of 0.6 percent, there is a wide diversity of stress-rupture life. One specimen failed in 236.2 hours (specimen 60) and the other in 620.4 hours (specimen 61). In the photomicrographs of the fractured specimens there is no evidence to explain the superior behavior of one specimen relative to the other. However, the photomicrographs before the test, show that the better specimen (specimen 61) had fewer and smaller residual carbides than the poorer one (specimen 60). Densities of the two specimens were similar and could not explain property differences.

Generalized Comments on Relations of

Microstructures to Properties

The methods of fabricating specimens used in this investigation resulted in variations in microstructures. Depending on slight, unavoidable variations in sintering time, incipient melting, specimen size, and degree of initial blending of alloy powder and carbon, two specimens of a given carbon content can exhibit different microstructures. At times, a specimen of a particular carbon content had a microstructure containing large grain-boundary carbides with little, if any, precipitation within the grains. At other times, a specimen having the identical carbon content had a microstructure containing small grain-boundary carbides and carbide precipitation within the grains. Microstructural variations such as these result not only in differences in strength properties in the as-sintered condition but also in differences in response to subsequent thermal and mechanical treatment. As a result, it is reasonable to expect that any scatter found in strength properties of as-sintered specimens of equivalent carbon content will also be seen even after these specimens receive working or heat treatments, unless these treatments are drastic enough to eliminate any initial microstructural differences that existed. The present work supports this statement. If photomicrographs of specimens of a given carbon content and history are compared, it can be seen that only those in the hot-swaged plus heat-treated group possess essentially identical microstructures. This fact is, in turn, reflected in the rupture lives of this group, which exhibit little scatter in comparison to those of the other groups tested.

Perhaps a more interesting result of microstructural variations is the fact that they permit evaluation of two types of strengthening,

strengthening due to grain-boundary carbides and that due to a more or less uniform dispersion of carbide precipitate within the grains. examinations of many pairs of specimens that had equivalent heat treatments or fabrications, there seemed to be an almost perfectly consistent indication that the stronger of the two specimens had fewer massive or residual carbides in the grain boundaries than the weaker of the two. exception to this generalization occurred in the case of the low-carbon, as-sintered specimens that not only had better stress-rupture lives when grain-boundary carbides were present but also generally exhibited longer lives than low-carbon specimens of other groups. This seems to indicate that a distribution of grain-boundary carbides is more effective in providing strengthening in low-carbon specimens than is precipitation uniformly distributed within grains. The effect is probably due to the prevention of grain-boundary sliding by the distributed carbides. these low-carbon specimens the grain-boundary carbides are removed or partially dissolved (as is the case in low-carbon specimens of the swaged, heat-treated, and swaged and heat-treated groups), the strength is low-This can be explained on the basis that any solid-solutionstrengthening or precipitation-strengthening that occurs as a result of the solution and elimination of large grain-boundary carbides is just not great enough to counterbalance the strength loss caused by removal or diminution of grain-boundary carbides. Even if all carbides were dissolved, the strengthening would probably be small simply because of the lack of sufficient available carbon in low-carbon specimens to provide efficient solution- or precipitation-strengthening. On the other hand, in higher carbon specimens, where there is an abundance of available carbon tied up as carbides, and the dissolution and subsequent dispersion of these carbides can lead to an increase in strength by solution- and/or dispersion-strengthening mechanisms that is greater than the initial strengthening afforded by grain-boundary carbides. Thus, in higher carbon specimens, the greater the degree of carbide dispersion or solution within grain (which is equivalent to a decrease in number and size of grain-boundary carbides), the stronger the specimen. Therefore, these results tend to show that, when it is not possible to achieve adequate precipitation- or solid-solution-strengthening, grain-boundary carbides or carbide networks may serve as effective alloy strengtheners.

Correlations of Ductility with Stress-Rupture

Life and Microstructure

Ductility plots made in figures 2 to 5 show peaks in ductility curves ranging from approximately 0.2 to 0.5 percent carbon. The position of the peak varies with the condition of the specimens. The curve for the as-sintered specimens peaks or has a maximum ductility value at less than 0.15 percent carbon; the ductility curve of the heat-treated specimens peaks at approximately 0.2 percent carbon; the ductility curve of the

swaged specimens peaks at approximately 0.5 percent carbon; and for the swaged and heat-treated specimens there is no sharp peak, but there is a plateau ranging from 0.2 to 0.6 percent carbon. In each case the ductility drops sharply with increasing carbon content after the maximum ductility has been obtained. This type of behavior is often observed in alloys.

The dropoffs in ductility to the left of the peaks or with decreasing carbon content below that of the peak are difficult to rationalize. The best evidence to indicate such a true dropoff is shown in figure 5, which represents the swaged and heat-treated specimens. A very significant dropoff in ductility, as represented by reduction in area, may be The ductility drops from 30 percent to about 7 percent. Although the left portions of these ductility curves were not established by large numbers of specimens in these regions, the fact that the dropoffs were observed to some degree in all curves indicates that such a dropoff is probably a true effect and not simply a coincidence. Studies of the microstructures of the low-carbon specimens relative to the others have not revealed a completely concrete reason for such a dropoff. as-sintered specimens, for example, none of the specimens with carbon contents less than 0.2 percent had ductility values below 19 percent. All the low-carbon specimens contained either some Chinese script or discontinuous or partial network structures, with the bulk of the grainboundary areas being free of continuous or thick structures. Typical photomicrographs of such specimens are shown in figure 10. Even in the tested conditions, most of the grain boundaries in the as-sintered specimens do not reveal any large amount of precipitation after testing. As the carbon contents are raised in these specimens, more general precipitation and residual carbides as well as more grain-boundary precipitation occurs, and all of these factors may contribute to the low ductility of the high-carbon specimens.

In general, no consistent pattern of microstructural differences was found to explain differing ductilities of specimens. There were indications that the low-carbon specimens that contained fairly extensive grain-boundary precipitations were relatively brittle, whereas low-carbon or even intermediate-carbon specimens that contained partially spheroidized or overaged carbides in the boundaries after testing exhibited higher ductilities. Where specimens remained in a solution condition throughout most of the test there appeared to be high ductilities. Where large numbers of carbides appeared, whether residual, or aged out during the test, specimens had relatively low ductilities. Generally speaking, another observation that might be made is that the lower carbon specimens had larger grain sizes than did the higher carbon specimens. The large grain size of the lower carbon specimens could be attributed to the sintering temperature necessary to cause incipient fusion of these specimens, which was higher than those needed for higher carbon specimens. Also, the

presence of more carbides would tend to inhibit grain growth in the higher carbon specimens.

Almost all the fracture edges showed intergranular fractures as well as intergranular stress-rupture cracks. In low-carbon specimens there was some evidence that the grains were distorted during the testing. Such distortions were evidenced by elongations of some of the grains and by slip-line precipitation in some of the grains. Upon examination, the fracture edges did not reveal any consistent explanation for the low ductility of either the low-carbon or the high-carbon specimens. It might be mentioned that some of the high-carbon specimens showed considerable ductility of the grains themselves.

One additional point that might be made is that, in all groups of specimens with the exception of the swaged group, measured ductility values of specimens of a given carbon content and treatment had reasonably equivalent ductilities whether or not the specimens had high or low stress-rupture values. In the case of the swaged specimens, however, as represented by figure 4, those specimens with high stress-rupture lives had low ductilities and those specimens with low rupture life had significantly high ductilities. No explanation is offered for this behavior.

Potential of Powder-Metallurgical Process

In order to keep the number of specimens to a reasonable number, only one swaging process and one heat-treating process were used. These were chosen to simulate commercial treatments used on S-816. A question arises, then, as to whether the properties of the alloy made by powder-metallurgy methods would be improved if different swaging and/or heat treatments had been used. This question, of course, cannot be answered without further experimentation. However, it is difficult to believe that the swaging treatment and heat treatment used in this investigation were optimums for every specimen, especially since the specimens varied in carbon content. For example, at 2150° F the plasticity of an alloy of one composition might be different from that of alloys of another composition. swaging at 2150° F might be more effective in closing pores of one specimen than another. In the same manner, the solution-treating temperature of 2150° F might be more effective for high-carbon specimens, whose melting points are closer to the solution temperature, than for low-carbon specimens, whose melting points are farther removed from the solution temperature.

Therefore, while the upper edge of the scatter bands shown in plots of stress-rupture life against carbon content indicates the potential of the powder-metallurgy process and treatments used in this investigation, it by no means can be considered an absolute limit.

CONCLUSIONS

This investigation was intended to determine the effect of carbon content and the effect of type of carbide formation and distribution on the stress-rupture properties and forgeability of S-816 type alloys. Specimens were prepared by powder-metallurgy methods and variations in carbide formations and distribution were effected by mechanical working, heat treatment, or combinations of both. The following conclusions were drawn:

- 1. The powder-metallurgy processes used in this investigation produced many specimens with good stress-rupture lives. The majority of specimens had lives longer than that of standard wrought S-816 tested under the same conditions (25,000 psi at 1500° F). For the most part, these specimens had higher carbon contents than the nominal 0.4 percent carbon found in standard wrought material, but several specimens of higher strength had carbon contents considerably lower than 0.4 percent.
- 2. Powder-metallurgy techniques resulted in highly workable structures. Even specimens containing as much as 1.0 percent carbon were successfully hot-swaged with a reduction in area of 50 percent.
- 3. Increasing the carbon content of specimens resulted in an increase in stress-rupture life regardless of the working or heat treatment given the specimens.
- 4. In the low-carbon-concentration range, as-sintered specimens had stress-rupture lives that were superior to those of specimens given additional working and/or heat treatment. At higher carbon contents a good comparison is more difficult because of the increasing spread of the scatter bands as the carbon content increases. However, as the carbon content increased, the curve of stress-rupture life against carbon content for the hot-swaged and heat-treated group rose more steeply than the same curve for other groups.
- 5. Correlation of microstructures with stress-rupture lives indicated that, when carbon contents were very low, a structure containing grain-boundary carbides was more effective in strengthening specimens than other types of structures. This result was probably due to the lack of available carbon in this range for effective solid-solution- or precipitation-strengthening. At higher carbon concentrations, solutioning of grain-boundary carbides with subsequent precipitation within the grains gave a stronger structure.
- 6. Appreciable scatter in rupture lives generally occurred in the as-sintered, heat-treated, or hot-swaged specimens. A combination treatment of hot-swaging followed by heat treatment minimized scatter. This

result can be attributed to a reduction in density variations from specimen to specimen as well as a homogenization of structure.

7. Ductility curves exhibited maximum values in the neighborhood of 0.2 to 0.5 percent carbon. Maximum values ranged from about 20 to 30 percent reduction in area. These values are high in comparison to those exhibited by wrought specimens.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, April 12, 1962

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TABLE I. - STRESS-RUPTURE PROPERTIES OF TEST SPECIMENS

Spec- imen	Treatment	Carbon, percent	Stress- rupture 11fe, hr	Ductility, percent reduction in area	Density, g/cc	Percentage of wrought alloy density ^a
1 2 3 4 5	As-sintered	0.015 .105 .10 .195 .26	42.8 88.55 51.4 55.6 Broke on loading	24.6 22.1 19.7 28.9 Broke on loading	8.76 8.45 8.60 8.59 7.24	102.0 98.4 100.1 100.0 84.3
6		.37	Broke on loading	Broke on loading	6.45	75.1
7		.45	Broke on loading	Broke on loading	7,16	83.3
8 9 10		.36 .42 .58	153.5 296.2 351.0	23.8 20.4 	7.93 7.68 7.96	92.4 89.4 92.7
11 12 13 14 15 16 17		.64 .74 .80 .76 .775 .995	226.9 185.9 182.3 272.0 322.6 485.0 264.3	15.2 12.5 11.8 10.8 10.8 11.1 7.43	8.21 8.54 8.71 8.41 8.39 8.39 8.44	95.6 98.4 101.5 97.9 97.8 97.8 98.3
18 19	Heat-treated	0.085	9.2 Broke on	Broke on	8.90 6.99	103.7 81.4
20 21 20		.16 .17 .245	loading 34.6 22.6 30.7	loading 21.2 18.5	8.95 8.84 8.36	104.1 103.0 97.3
23 24		b.3	45,8 Broke on loading	20.4 Broke on loading	8.71 6.74	101.2 78.4
25		b.4	Broke on loading	Broke on loading	7.39	86.1
26 27		.42	64.8 6.9	15.9 9.0	8.48 8.75	98.8 101.8
28 29 30 31 32 33		.56 .565 .66 .625 .80	58.4 210.45 126.8 113.6 103.6 657.0	12.5 12.7 12.5 11.7 7.74 5.66	8.37 8.65 8.61 8.19 8.13 8.72	97.6 100.5 100.1 95.4 94.7 101.5
34 35 36 37 38	Hot-swaged	0.02 .10 .11 .17	29.3 59.2 22.3 46.6 60.7	8.5 15.1 16.2 26.0 6.78	8.46 8.60 7.78 8.35 8.33	98.5 100.0 90.6 97.2 97.0
39 40 41 42 43		.185 .275 .355 .41 .61	38.8 40.2 16.8 21.4 176.9	36.2 20.8 30.8 6.78 19.0	8.31 8.21 8.67 8.50 8.40	96.9 95.6 101.0 98.9 97.9
44 45 46 47		.73 .735 .91 1.0	208.2 126.0 445.2 523.3	13.0 29.8 6.2 3.91	7.93 8.89 8.68 8.57	92.4 103.4 101.0 99.8
48 49 50 51 52	Hot-swaged and heat-treated	0.015 .08 .10 .15 .17	18.7 143.5 101.4 41.8 49.4	6.78 11.7 15.3 29.6 28.5	8.19 8.61 8.59 8.55 8.68	95.3 100.1 100.0 89.5 101.1
53 54 55 56 57		.18 .24 .31 .47	35.3 43.9 69.6 114.2 211.1	30.3 27.1 29.4 28.2 33.9	8.23 8.48 8.27 8.56 8.37	95.8 98.7 96.2 99.6 97.3
58 59 60 61		.55 .60 .64 .65	252.8 216.3 236.2 620.4	29.4 27.2 30.5 13.9	8.61 8.71 8.46 8.57	100.1 101.2 98.3 99.8

^{8.59} g/cc.

b_{Nominal value.}

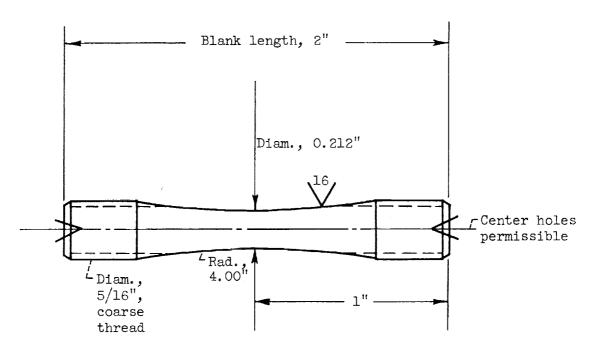


Figure 1. - Test specimen.

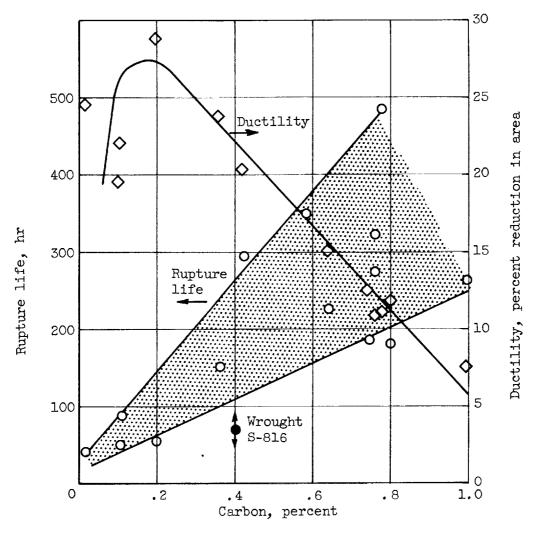


Figure 2. - Influence of carbon content on rupture life and ductility of powder-metallurgy S-816 in as-sintered condition. Stress, 25,000 pounds per square inch; temperature, 1500° F.

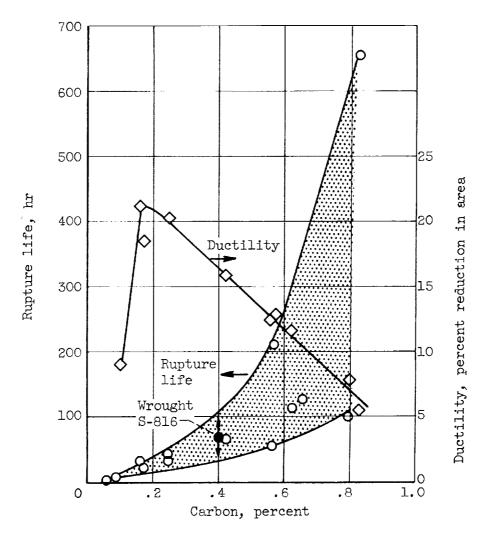


Figure 3. - Influence of carbon content on rupture life and ductility of powdermetallurgy S-816 after heat treatment (24 hr at 2150° F, water-quenched, aged 16 hr at 1400° F, air-cooled). Stress, 25,000 pounds per square inch; temperature, 1500° F.

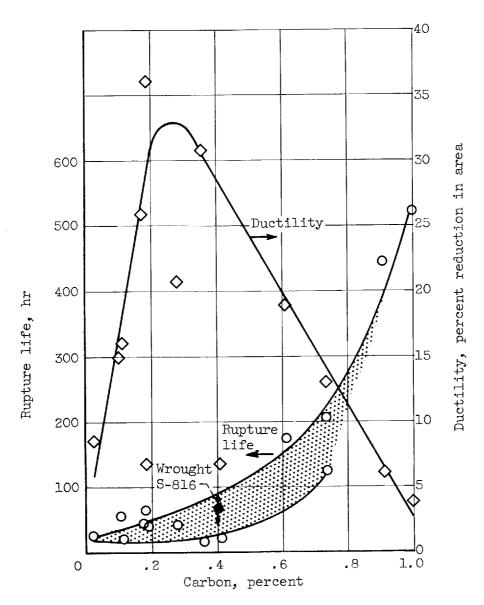


Figure 4. - Influence of carbon content on rupture life and ductility of powder-metallurgy S-816 after hot-swaging (at 2150° F to 50 percent reduction in area). Stress, 25,000 pounds per square inch; temperature, 1500° F.

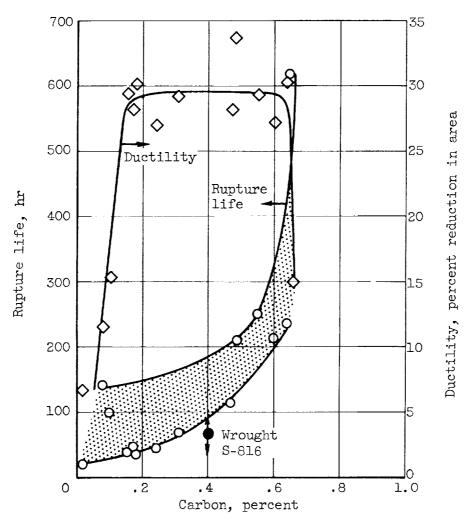
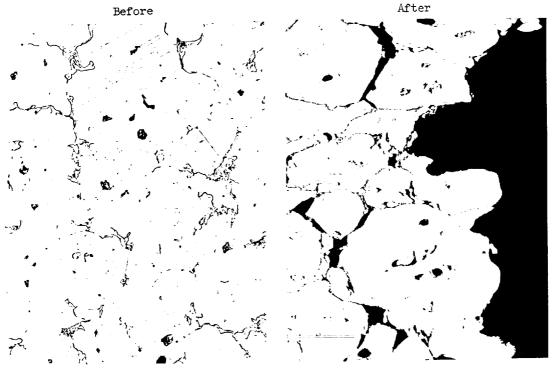
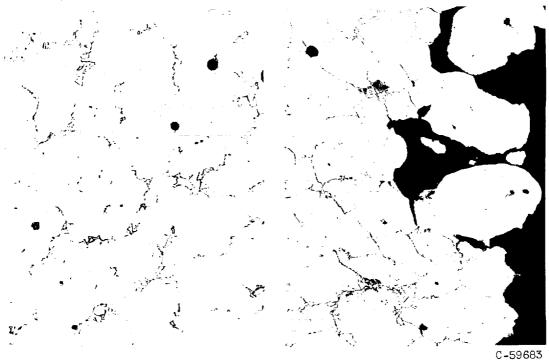


Figure 5. - Influence of carbon content on rupture life and ductility of powder-metallurgy S-816 after hot-swaging and heat-treatment. Stress, 25,000 pounds per square inch; temperature, 1500° F.

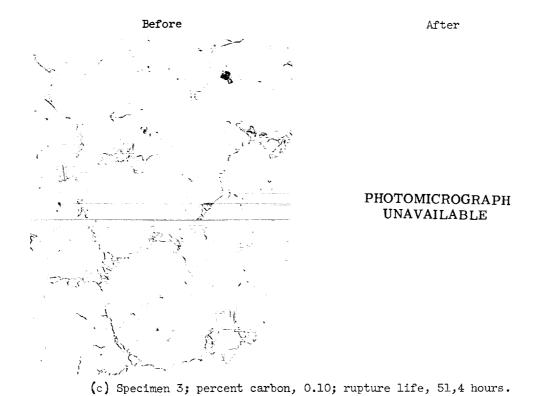


(a) Specimen 1; percent carbon, 0.105; rupture life, 42.8 hours.



(b) Specimen 2; percent carbon, 0.105; rupture life, 88.55 hours.

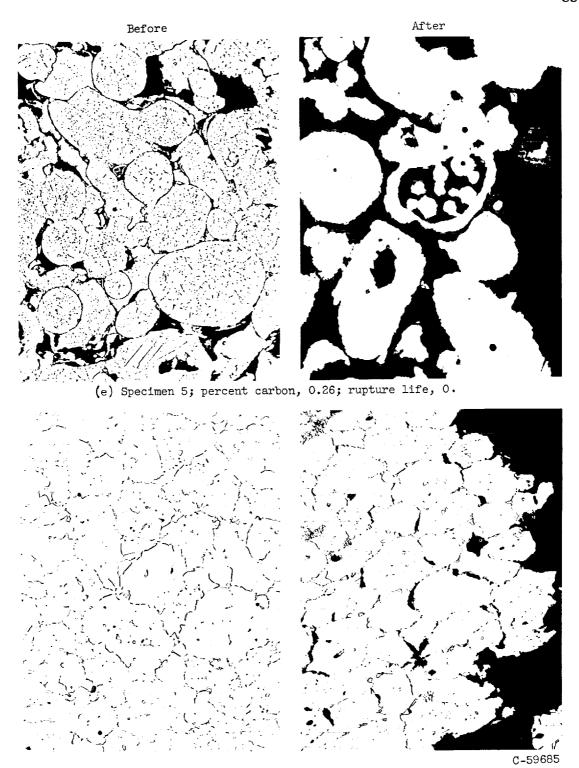
Figure 6. - Photomicrographs of as-sintered specimens before and after stress-rupture test. X250.



(d) Specimen 4; percent carbon, 0.195; rupture life, 55.6 hours.

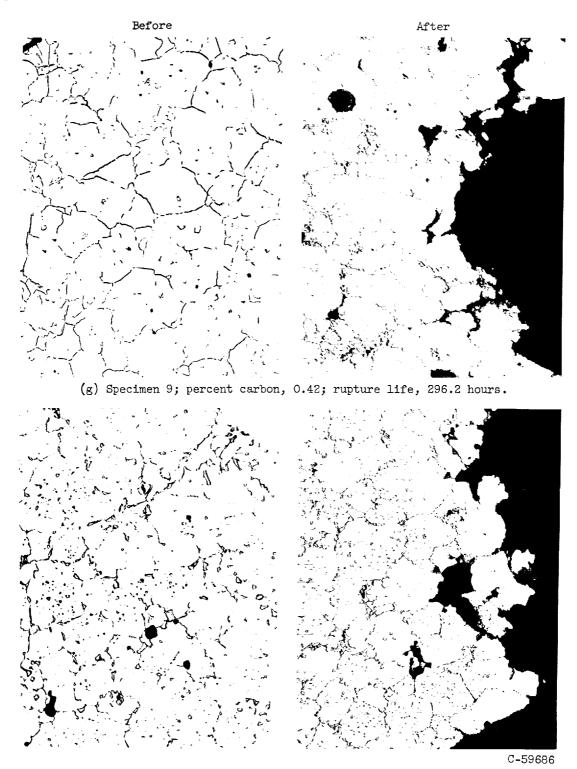
C-59684

Figure 6. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X250.



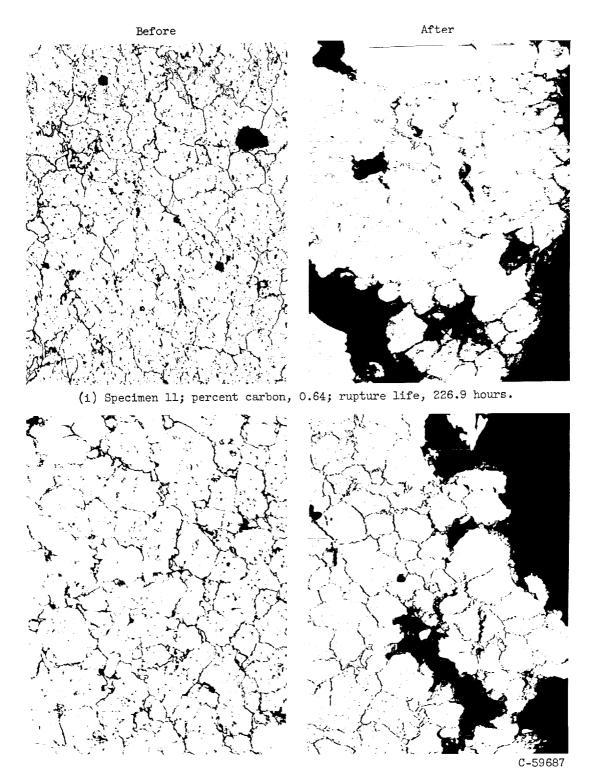
(f) Specimen 8; percent carbon, 0.36; rupture life, 153.5 hours.

Figure 6. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X250.



(h) Specimen 10; percent carbon, 0.58; rupture life, 351.0 hours.

Figure 6. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X250.



(j) Specimen 12; percent carbon, 0.74; rupture life, 185.9 hours.

Figure 6. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X250.

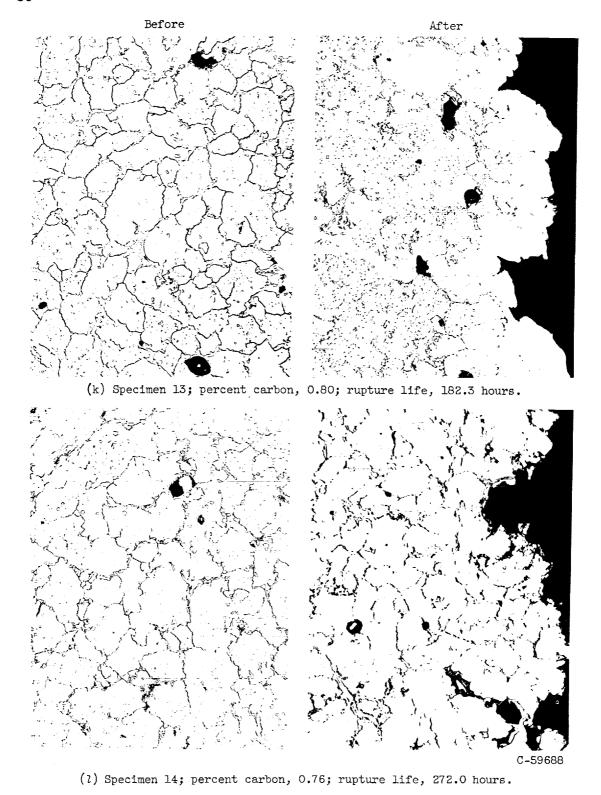
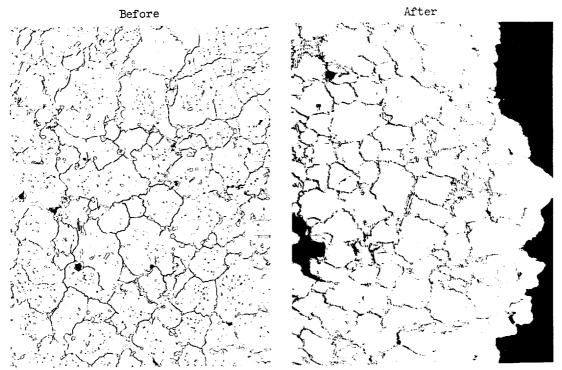
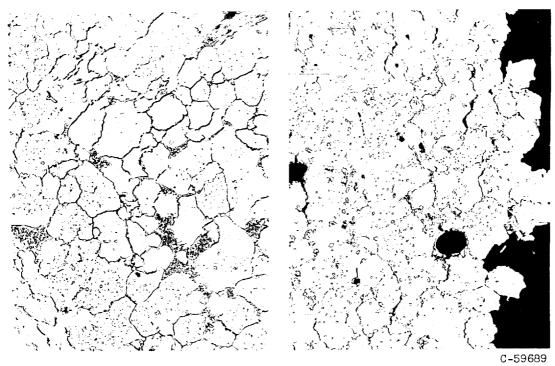


Figure 6. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X250.

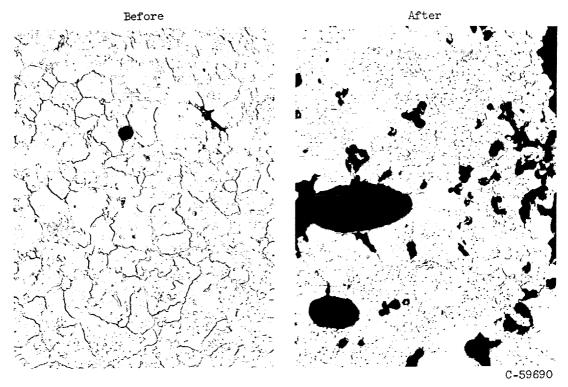


(m) Specimen 15; percent carbon, 0.76; rupture life, 322.6 hours.



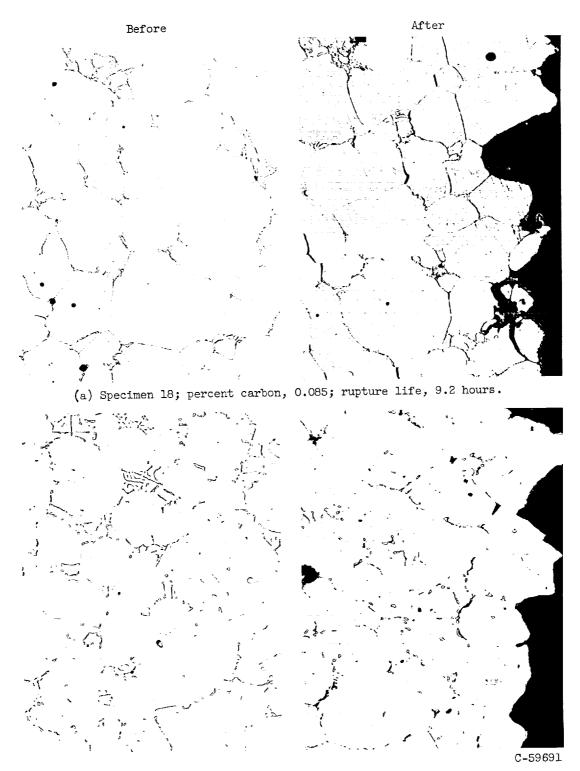
(n) Specimen 16; percent carbon, 0.775; rupture life, 485.0 hours.

Figure 6. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X250.



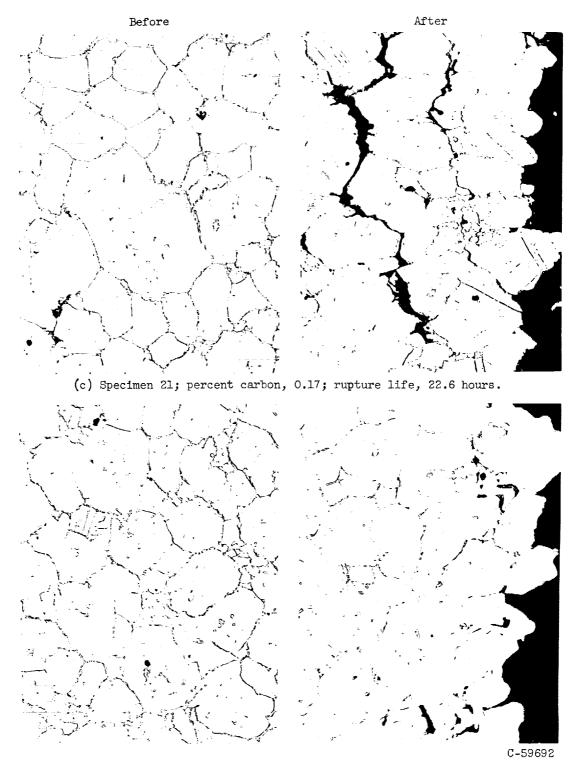
(o) Specimen 17; percent carbon, 0.995; rupture life, 264.3 hours.

Figure 6. - Concluded. Photomicrographs of as-sintered specimens before and after stress-tupture test. X250.



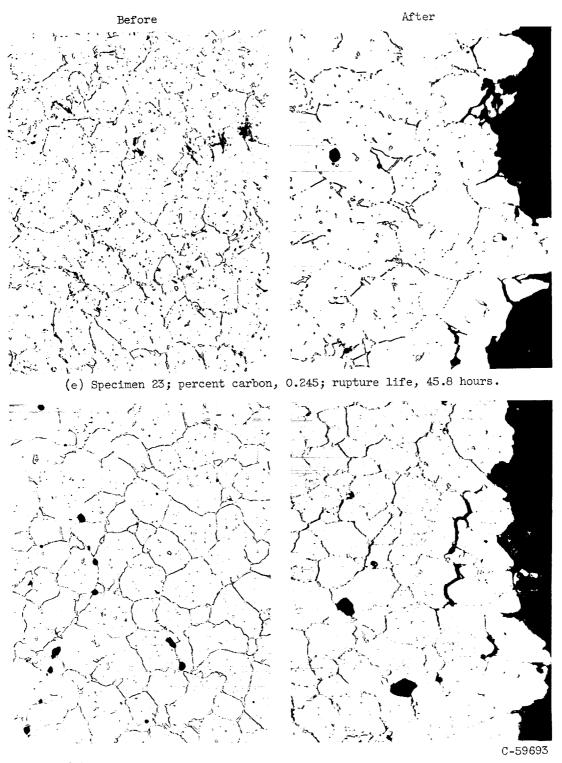
(b) Specimen 20; percent carbon, 0.16; rupture life, 34.6 hours.

Figure 7. - Photomicrographs of heat-treated specimens before and after stress-rupture test. X250.



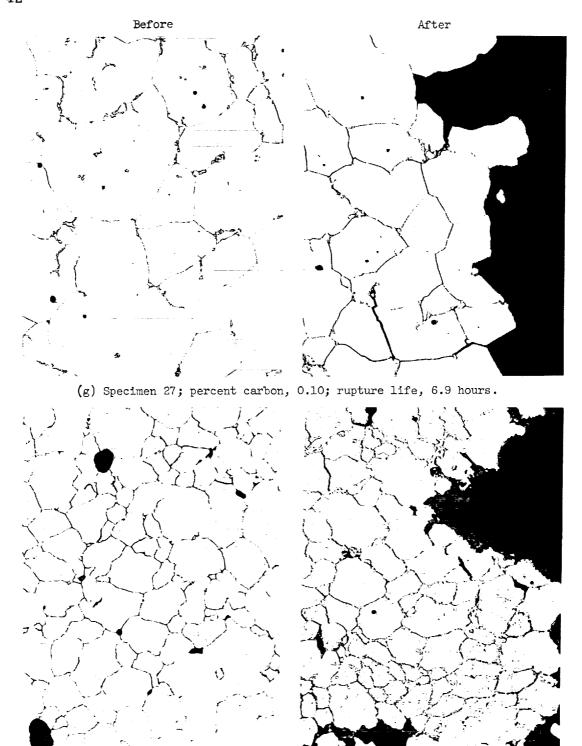
(d) Specimen 22; percent carbon, 0.245; rupture life, 30.7 hours.

Figure 7. - Continued. Photomicrographs of heat-treated specimens before and after stress-rupture test. X250.



(f) Specimen 26; percent carbon, 0.42; rupture life, 64.8 hours.

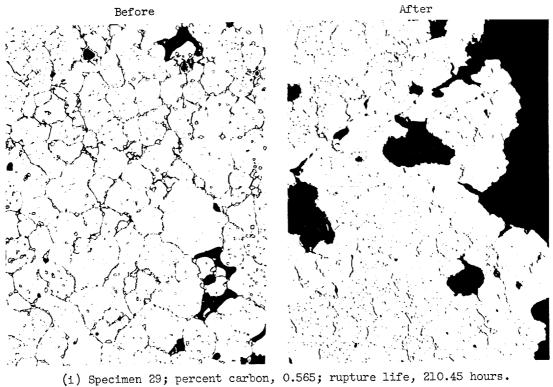
Figure 7. - Continued. Photomicrographs of heat-treated specimens before and after stress-rupture test. X250.

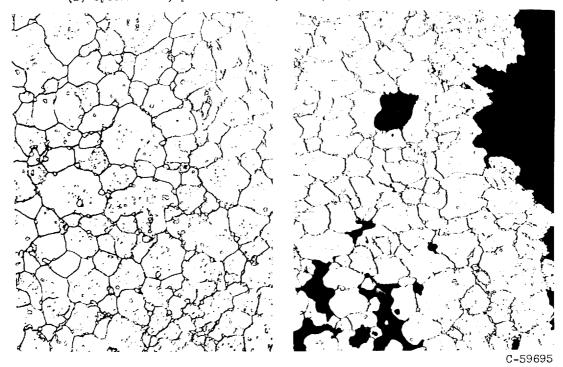


(h) Specimen 28; percent carbon, 0.56; rupture life, 58.4 hours.

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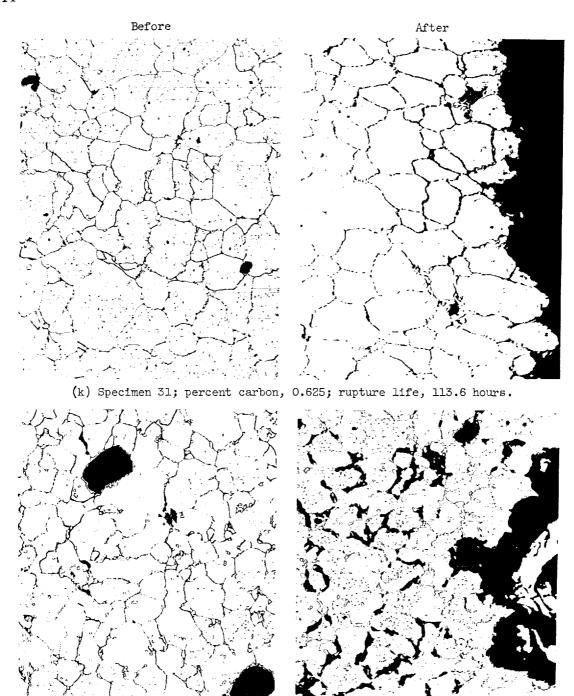
Figure 7. - Continued. Photomicrographs of heat-treated specimens before and after stress-rupture test. X250.





(j) Specimen 30; percent carbon, 0.66; rupture life, 126.8 hours.

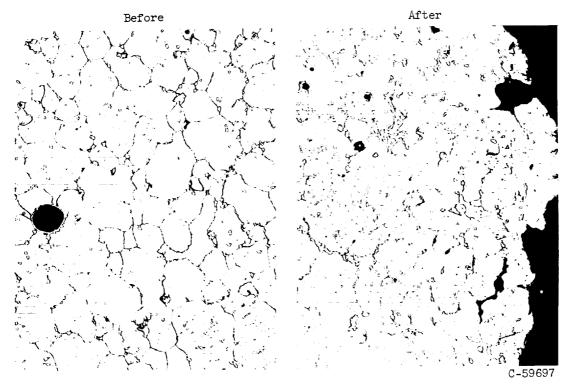
Figure 7. - Continued. Photomicrographs of heat-treated specimens before and after stress-rupture test. X250.



(1) Specimen 32; percent carbon, 0.80; rupture life, 103.6 hours.

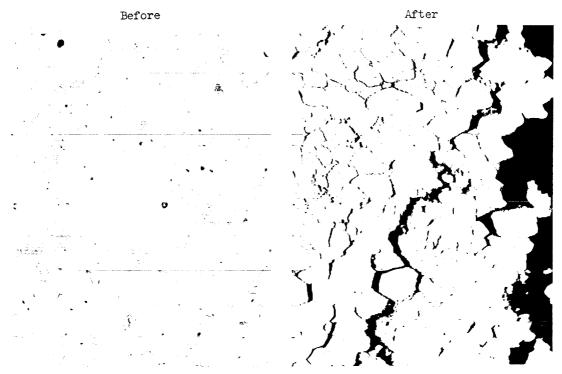
C-59696

Figure 7. - Continued. Photomicrographs of heat-treated specimens before and after stress-rupture test. X250.

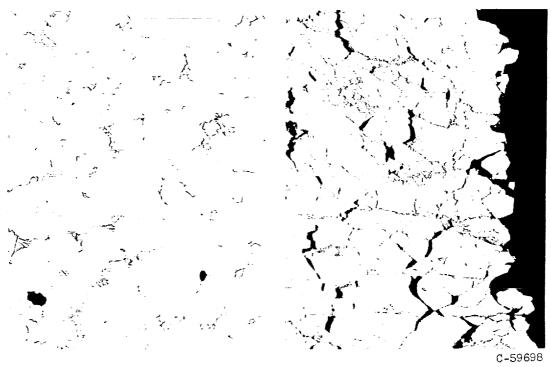


(m) Specimen 33; percent carbon, 0.835; rupture life, 657.0 hours.

Figure 7. - Concluded. Photomicrographs of heat-treated specimens before and after stress-rupture test. X250.

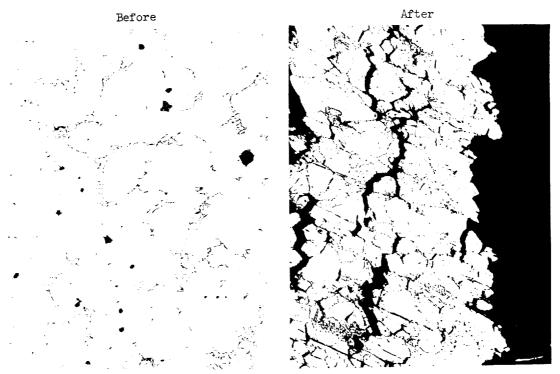


(a) Specimen 34; percent carbon, 0.02; rupture life, 29.3 hours.

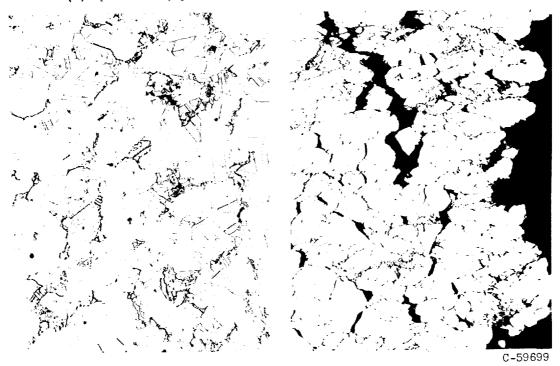


(b) Specimen 35; percent carbon, 0.10; rupture life, 59.2 hours.

Figure 8. - Photomicrographs of hot-swaged specimens before and after stress-rupture test. X250.

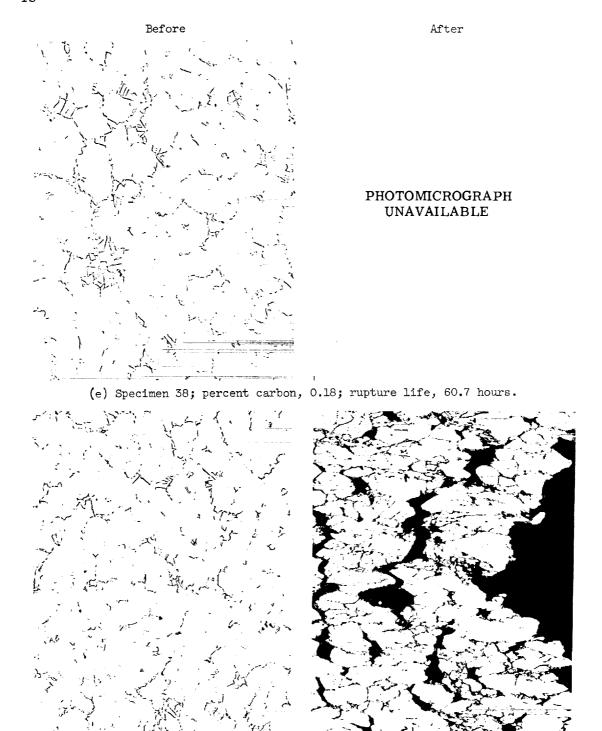


(c) Specimen 36; percent carbon, 0.11; rupture life, 22.3 hours.



(d) Specimen 37; percent carbon, 0.17; rupture life, 46.6 hours.

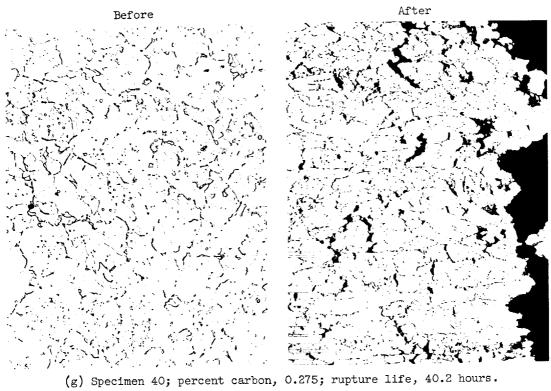
Figure 8. - Continued. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X250.

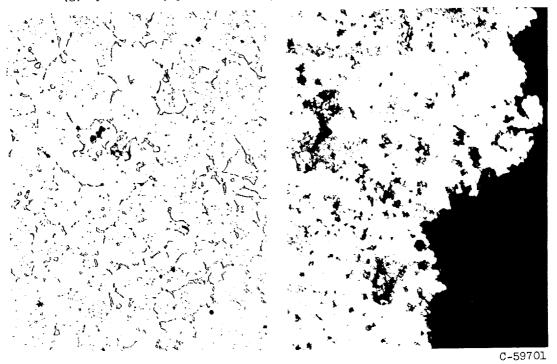


(f) Specimen 39; percent carbon, 0.185; rupture life, 38.8 hours.

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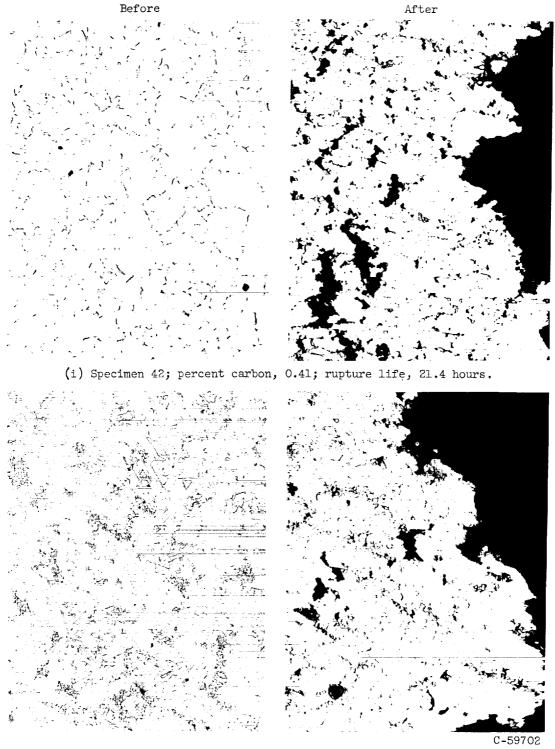
Figure 8. - Continued. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X250.





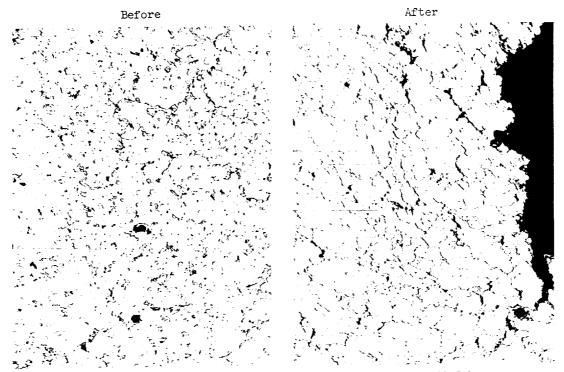
(h) Specimen 41; percent carbon, 0.355; rupture life, 16.8 hours.

Figure 8. - Continued. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X250.

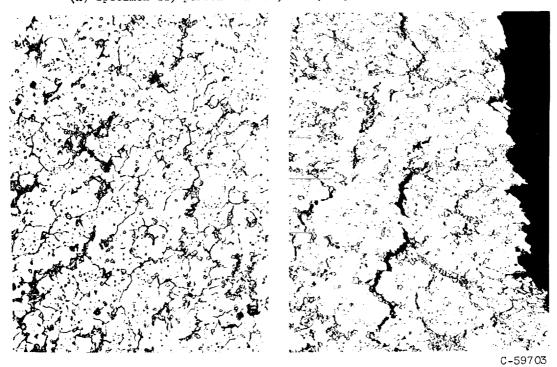


(j) Specimen 43; percent carbon, 0.61; rupture life, 176.9 hours.

Figure 8. - Continued. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X250.

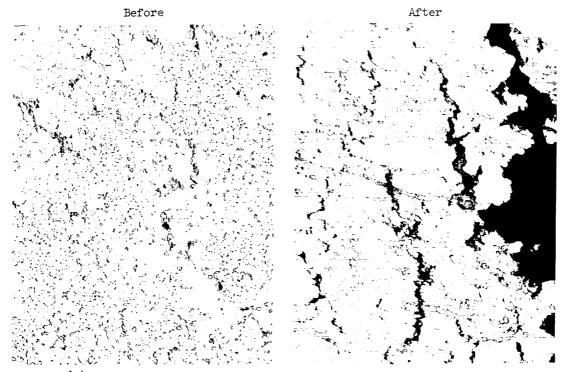


(k) Specimen 44; percent carbon, 0.73; rupture life, 208.2 hours.

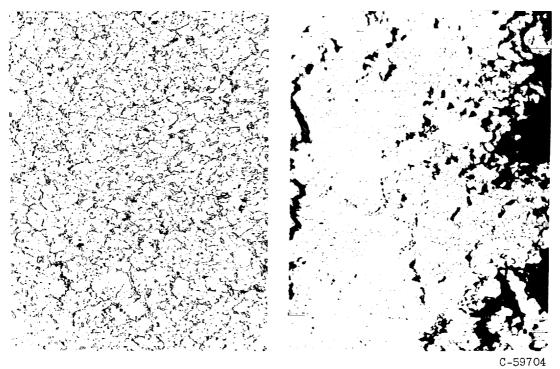


(1) Specimen 45; percent carbon, 0.735; rupture life, 126.0 hours.

Figure 8. - Continued. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X250.

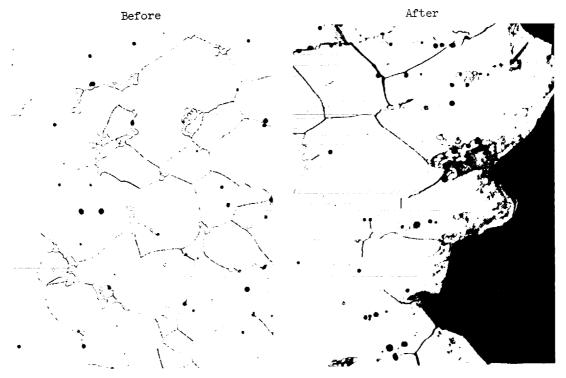


(m) Specimen 46; percent carbon, 0.91; rupture life, 445.2 hours.

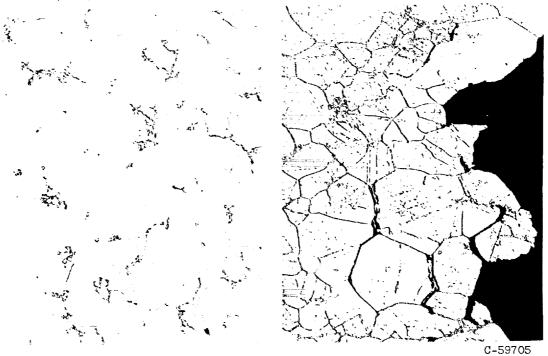


(n) Specimen 47; percent carbon, 1.0; rupture life, 523.3 hours.

Figure 8. - Concluded. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X250.

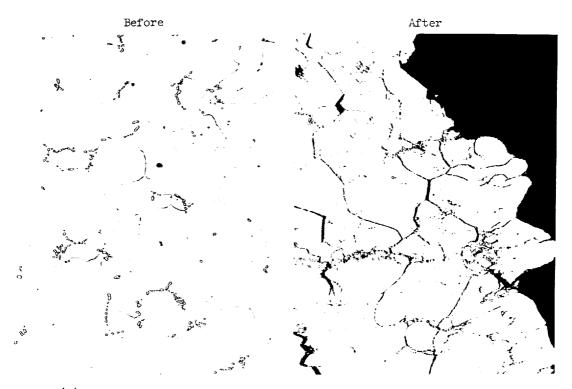


(a) Specimen 48; percent carbon, 0.015; rupture life, 18.7 hours.

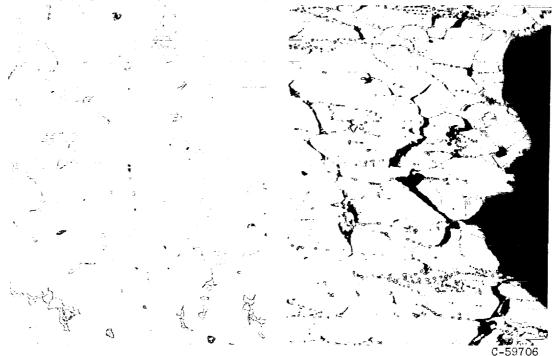


(b) Specimen 49; percent carbon, 0.08; rupture life, 143.5 hours.

Figure 9. - Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X250.

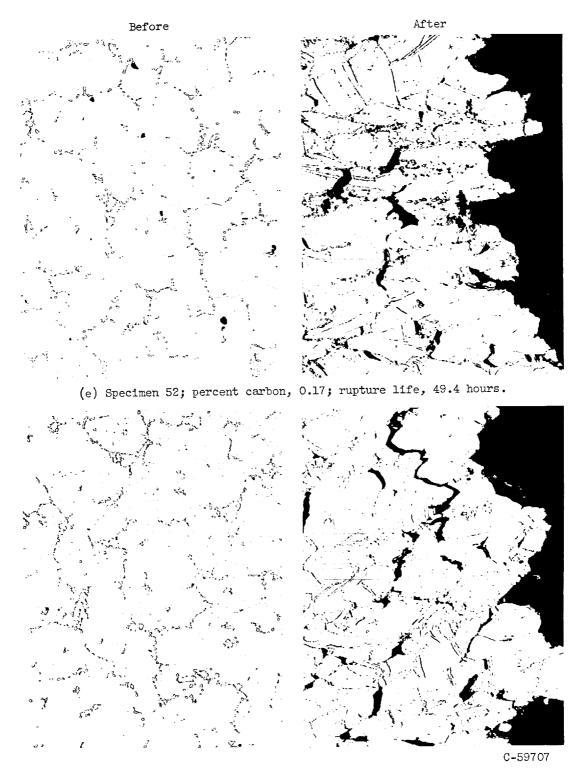


(c) Specimen 50; percent carbon, 0.10; rupture life, 101.4 hours.



(d) Specimen 51; percent carbon, 0.15; rupture life, 41.8 hours.

Figure 9. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X250.



(f) Specimen 53; percent carbon, 0.18; rupture life, 35.3 hours.

Figure 9. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X250.

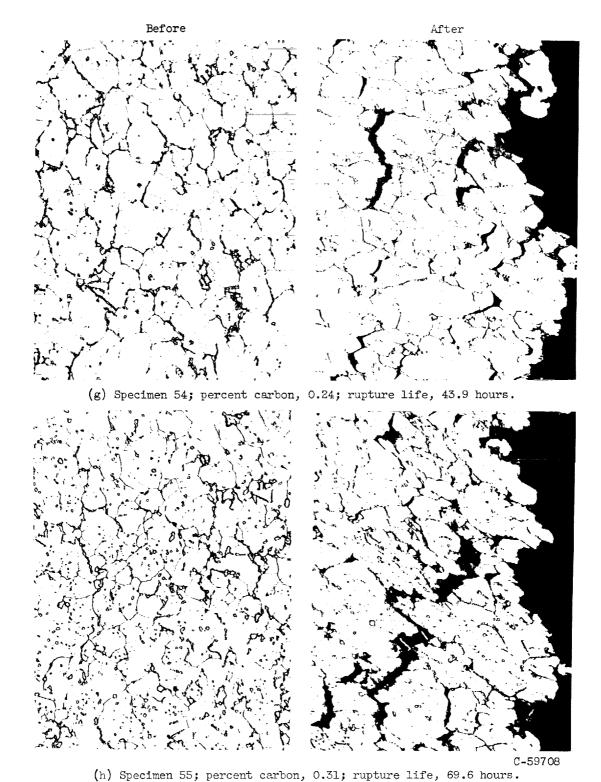
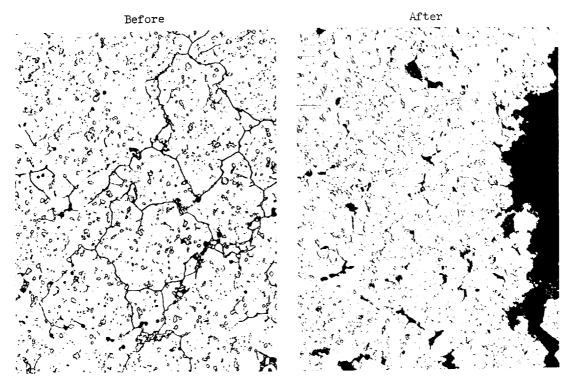
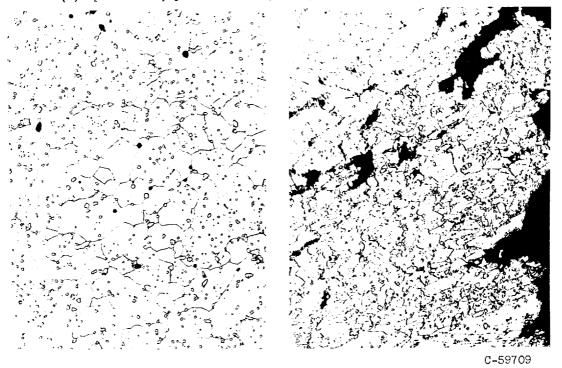


Figure 9. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X250.



(i) Specimen 56; percent carbon, 0.47; rupture life, 114.2 hours.



(j) Specimen 57; percent carbon, 0.49; rupture life, 211.1 hours.

Figure 9. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X250.

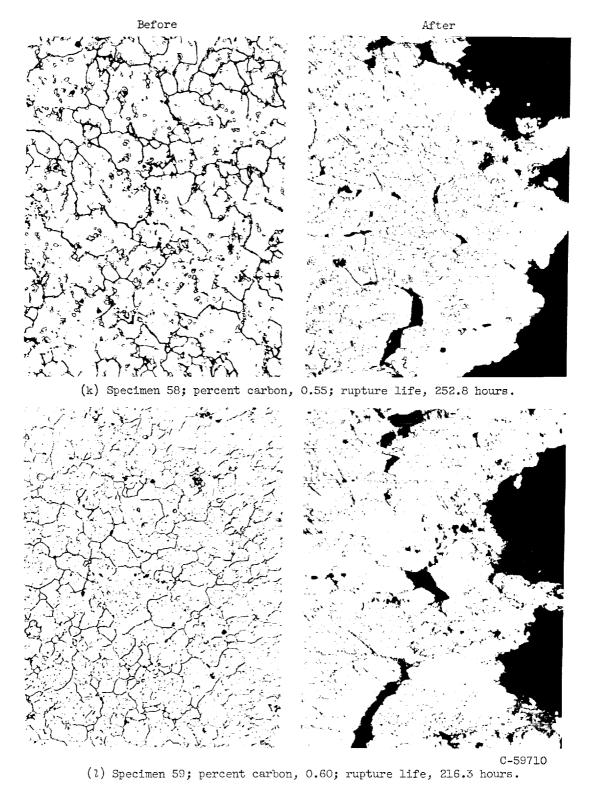


Figure 9. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X250.

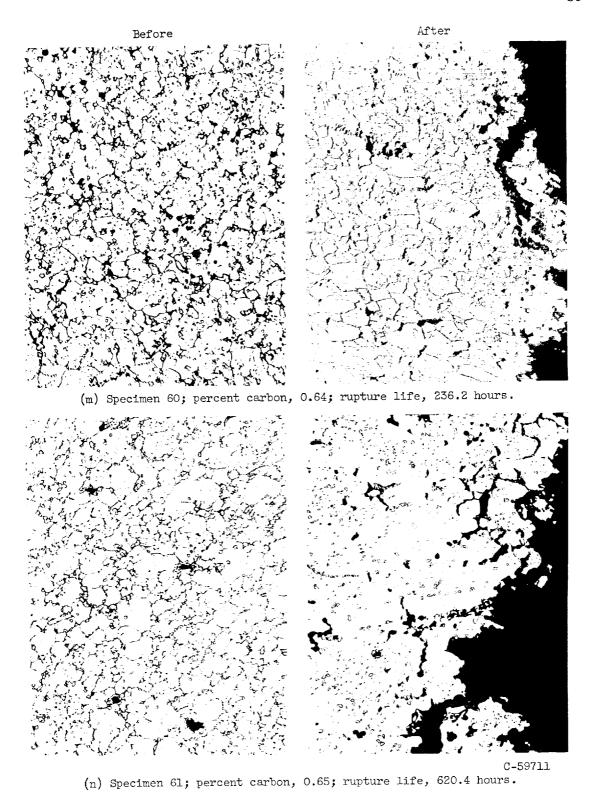
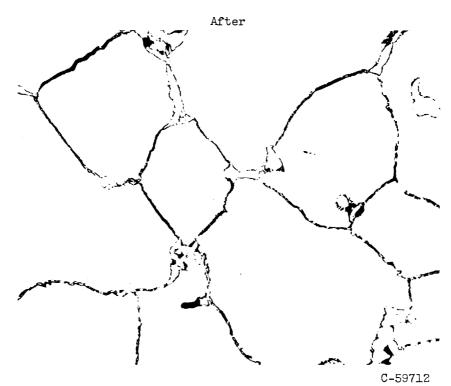
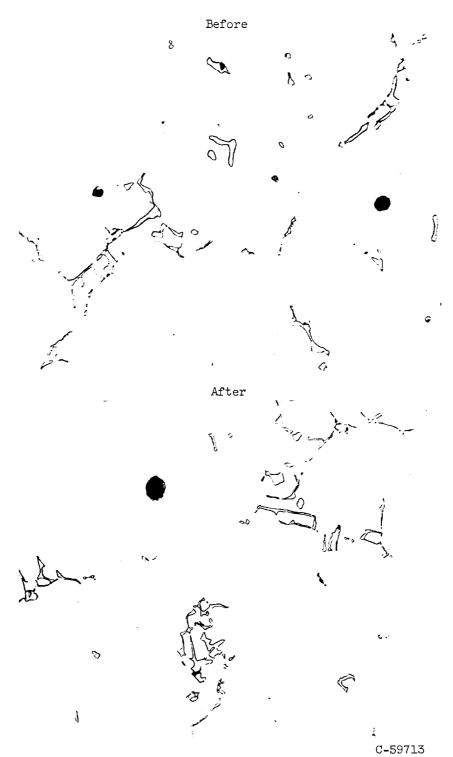


Figure 9. - Concluded. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X250.



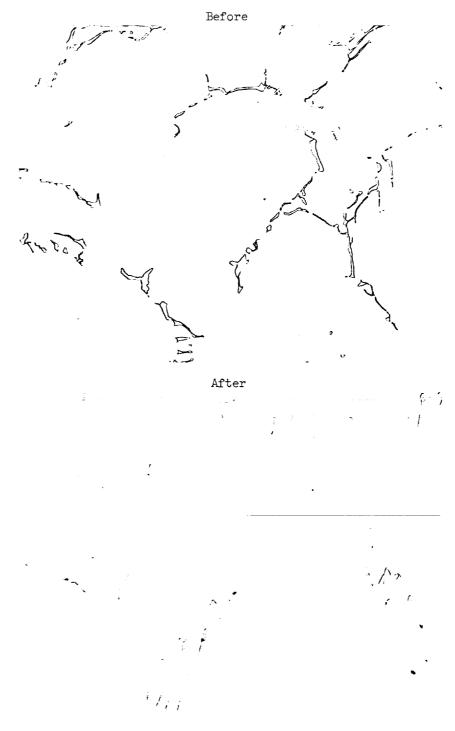
(a) Specimen 1; percent carbon, 0.015; stress-rupture life, 42.8 hours.

Figure 10. - Photomicrographs of as-sintered specimens before and after stress-rupture test. X750.



(b) Specimen 2; percent carbon, 0.105; stress-rupture life, 88.55 hours.

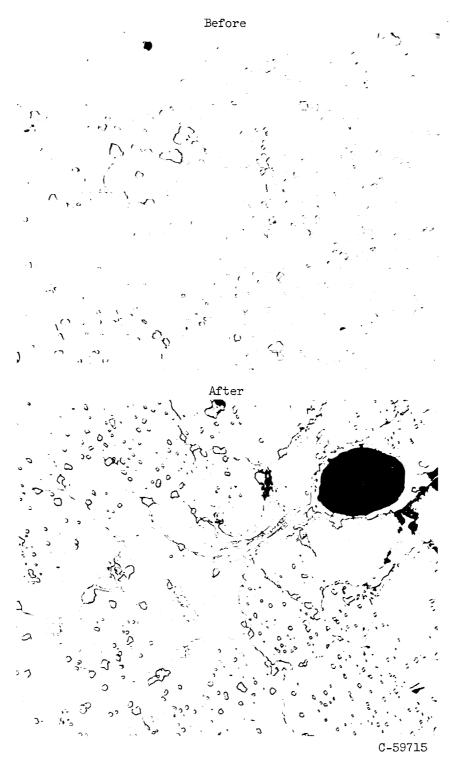
Figure 10. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X750.



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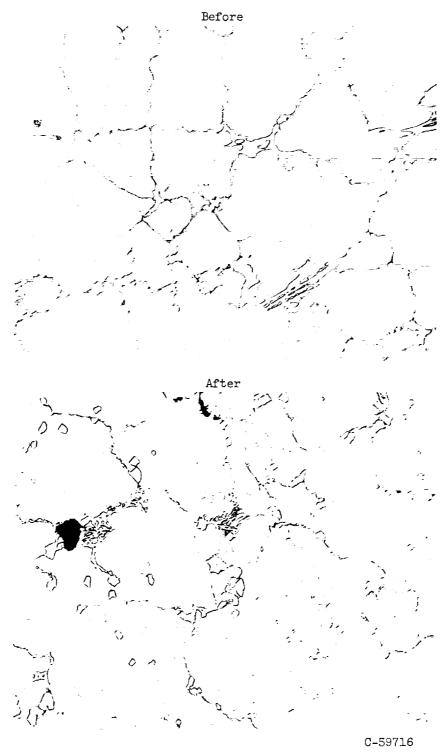
(c) Specimen 3; percent carbon, 0.10; stress-rupture life, 51.4 hours.

Figure 10. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X750.



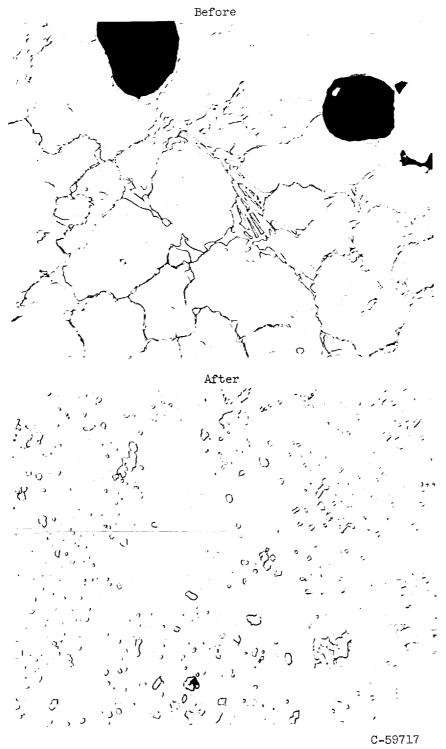
(d) Specimen 13; percent carbon, 0.80; stress-rupture life, 182.3 hours.

Figure 10. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X750.



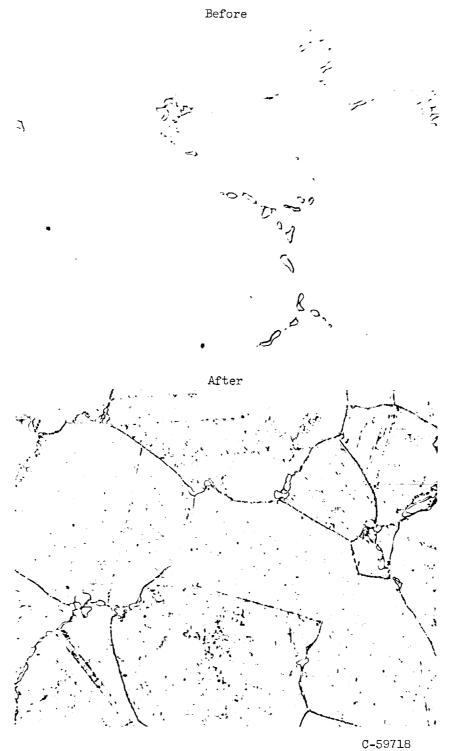
(e) Specimen 16; percent carbon, 0.775; stress-rupture life, 485.0 hours.

Figure 10. - Continued. Photomicrographs of as-sintered specimens before and after stress-rupture test. X750.



(f) Specimen 17; percent carbon, 0.995; stress-rupture life, 274.3 hours.

Figure 10. - Concluded. Photomicrographs of as-sintered specimens before and after stress-rupture test. X750.



(a) Specimen 18; percent carbon, 0.085; stress-rupture life, 9.2 hours.

Figure 11. - Photomicrographs of heat-treated specimens before and after stress-rupture test. X750.



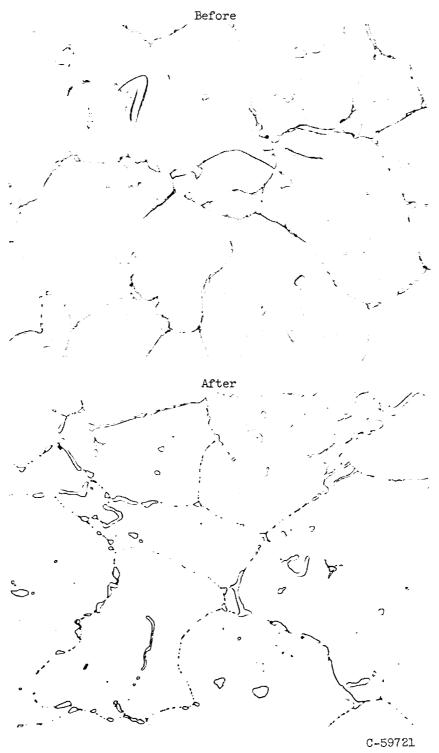
(b) Specimen 20; percent carbon, 0.16; stress-rupture life, 34.6 hours.

Figure 11. - Continued. Photomicrographs of heat-treated specimens before and after stress-rupture test. X750.



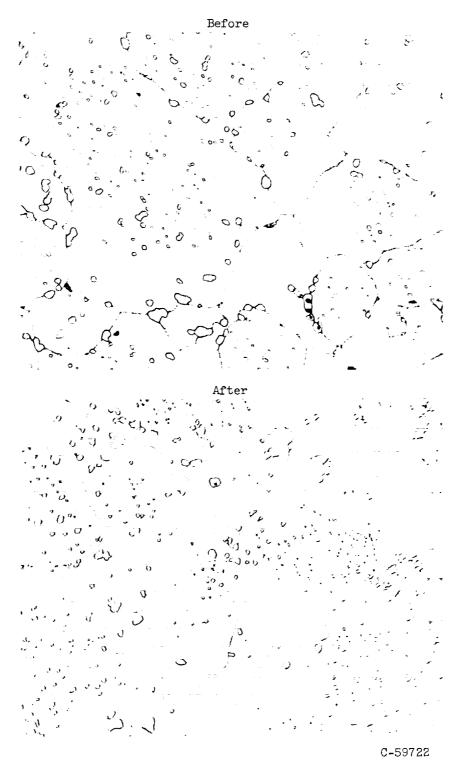
(c) Specimen 21; percent carbon, 0.17; stress-rupture life, 18.5 hours.

Figure 11. - Continued. Photomicrographs of heat-treated specimens before and after stress-rupture test. X750.



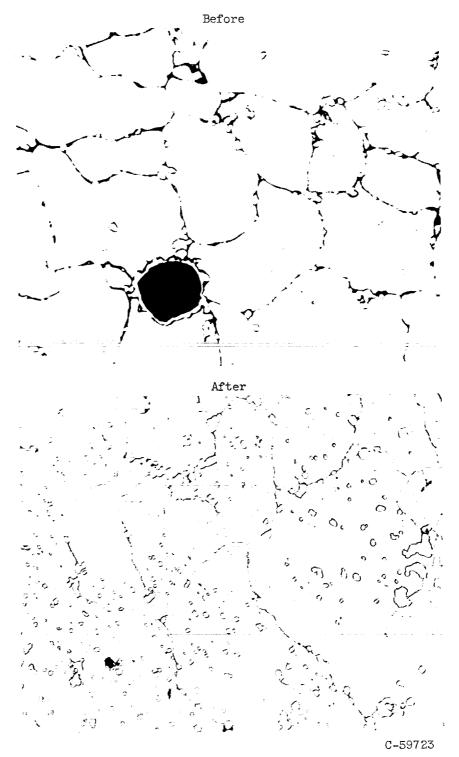
(d) Specimen 23; percent carbon, 0.245; stress-rupture life, 45.8 hours.

Figure 11. - Continued. Photomicrographs of heat-treated specimens before and after stress-rupture test. X750.



(e) Specimen 32; percent carbon, 0.80; stress-rupture life, 103.6 hours.

Figure 11. - Continued. Photomicrographs of heat-treated specimens before and after stress-rupture test. X750.



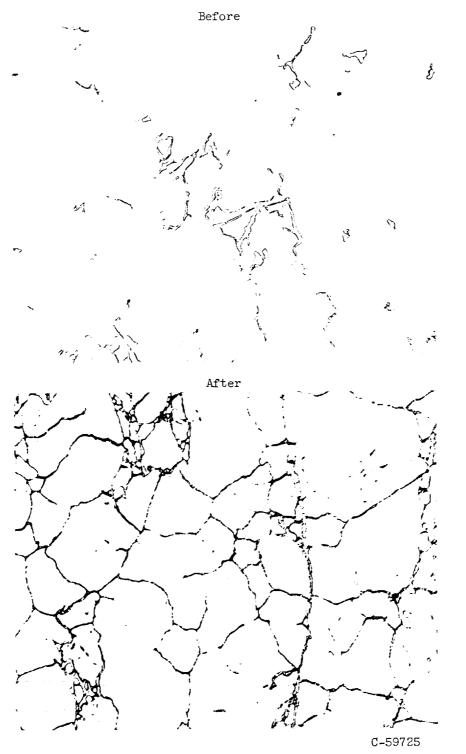
(f) Specimen 33; percent carbon, 0.835; stress-rupture life, 5.66 hours.

Figure 11. - Concluded. Photomicrographs of heat-treated specimens before and after stress-rupture test. X750.



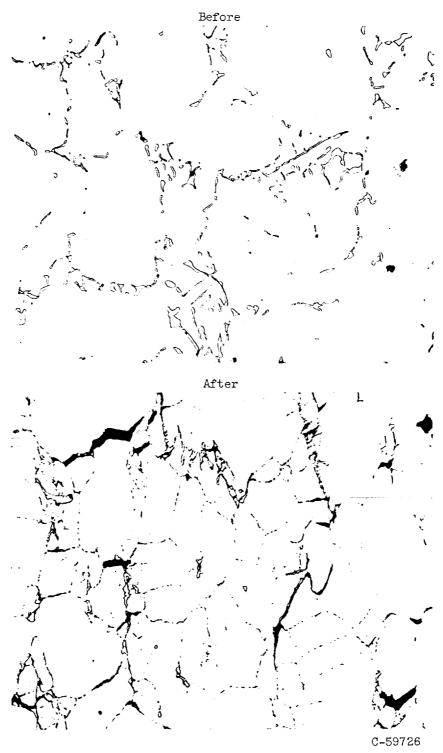
(a) Specimen 34; percent carbon, 0.02; stress-rupture life, 29.3 hours.

Figure 12. - Photomicrographs of hot-swaged specimens before and after stress-rupture test. X750.



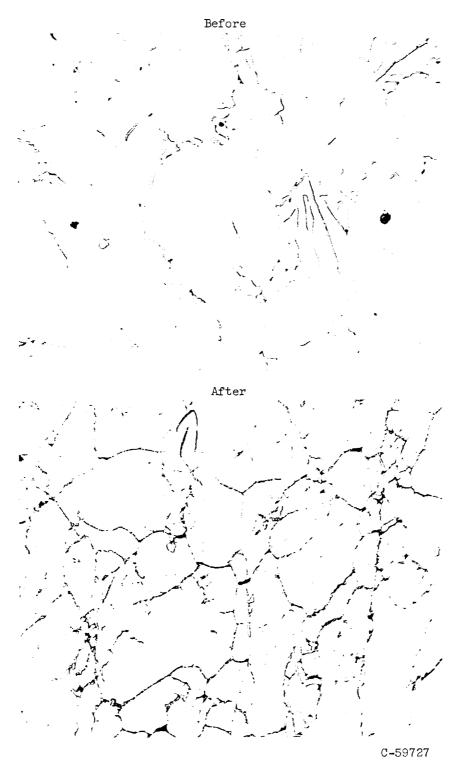
(b) Specimen 35; percent carbon 0.10; stress-rupture life, 59.2 hours.

Figure 12. - Continued. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X750.



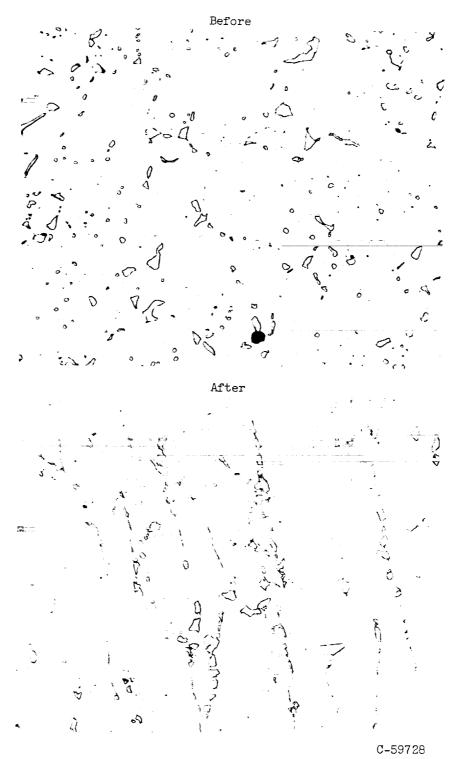
(c) Specimen 38; percent carbon, 0.18; stress-rupture life, 60.7 hours.

Figure 12. - Continued. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X750.



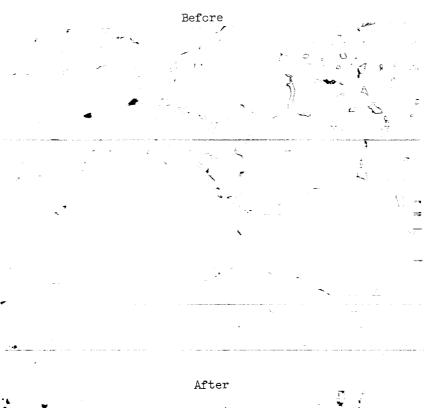
(d) Specimen 39; percent carbon, 0.185; stress-rupture life, 38.8 hours.

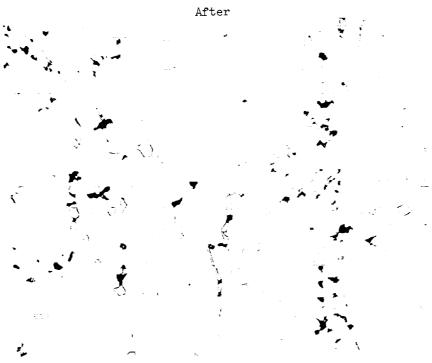
Figure 12. - Continued. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X750.



(e) Specimen 40; percent carbon, 0.275; stress-rupture life, 40.2 hours.

Figure 12. - Continued. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X750.





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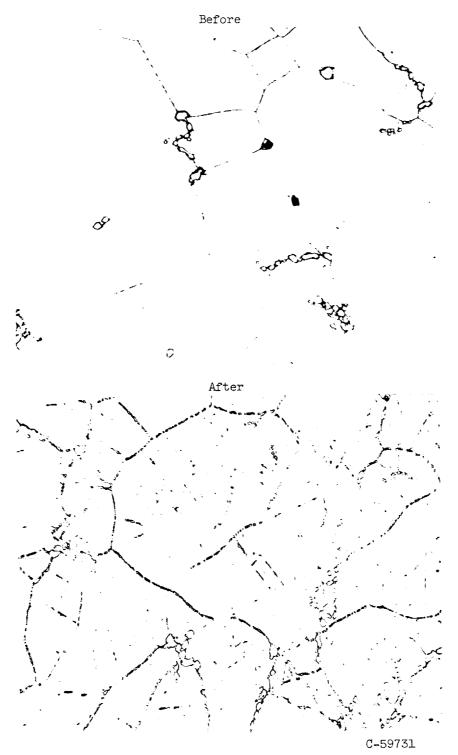
(f) Specimen 41; percent carbon, 0.355; stress-rupture life, 16.8 hours.

Figure 12. - Concluded. Photomicrographs of hot-swaged specimens before and after stress-rupture test. X750.



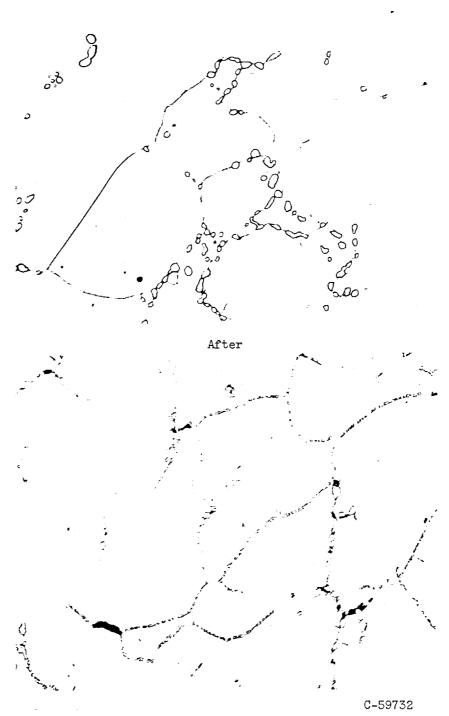
(a) Specimen 48; percent carbon, 0.015; stress-rupture life, 18.7 hours.

Figure 13. - Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X750.



(b) Specimen 49; percent carbon, 0.08; stress-rupture life, 143.5 hours.

Figure 13. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X750.



(c) Specimen 50; percent carbon, 0.10; stress-rupture life, 101.4 hours.

Figure 13. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X750.



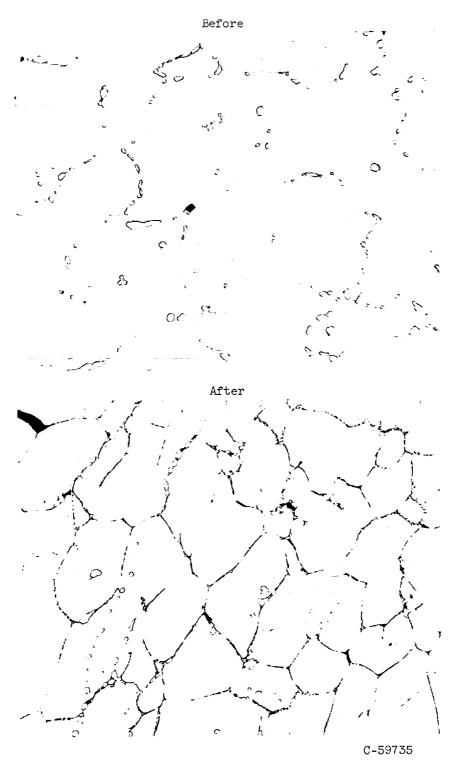
(d) Specimen 52; percent carbon, 0.17; stress-rupture life, 49.4 hours.

Figure 13. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X750.



(e) Specimen 53; percent carbon, 0.18; stress-rupture life, 35.3 hours.

Figure 13. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X750.



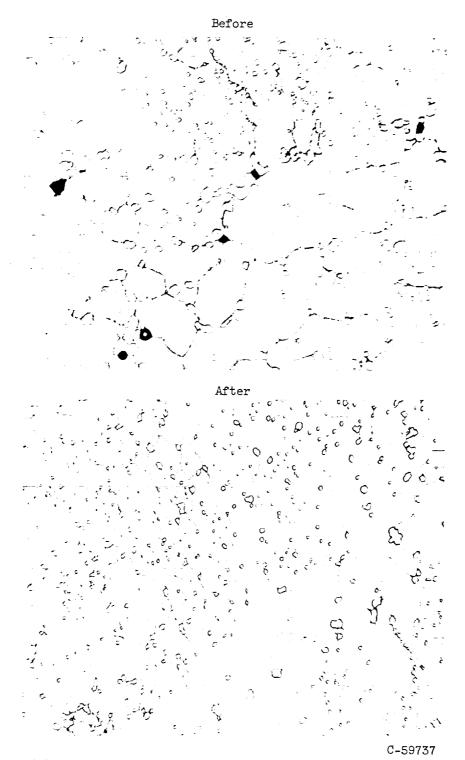
(f) Specimen 54; percent carbon, 0.24; stress-rupture life, 43.9 hours.

Figure 13. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X750.



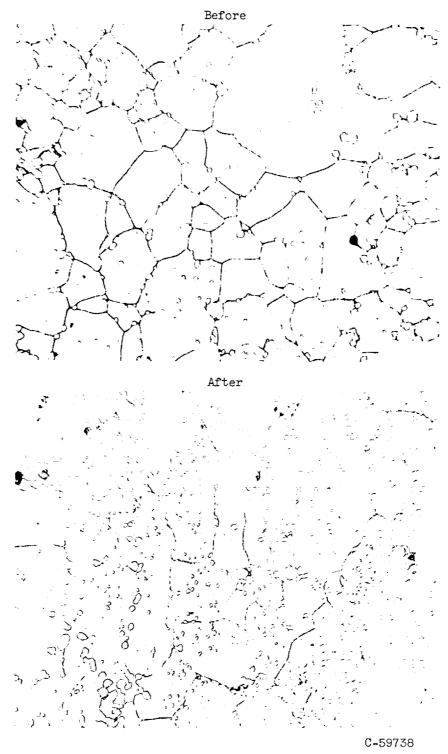
(g) Specimen 55; percent carbon, 0.31; stress-rupture life, 69.6 hours.

Figure 13. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X750.



(h) Specimen 60; percent carbon, 0.64; stress-rupture life, 236.2 hours.

Figure 13. - Continued. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X750.



(i) Specimen 61; percent carbon, 0.65; stress-rupture life, 620.4 hours.

Figure 13. - Concluded. Photomicrographs of hot-swaged and heat-treated specimens before and after stress-rupture test. X750.

NASA-Langley, 1962 E-149

I. Clarkin, Philip A. II. Weeton, John W. III. Sikora, Paul F. IV. NASA TN D-1221 (Initial NASA distribution: 25, Materials, engineering; 31, Physics, nuclear and particle.)	I. Clarkin, Philip A. II. Weeton, John W. III. Sikora, Paul F. IV. NASA TN D-1221 (Initial NASA distribution: 25, Materials, engineering; 31, Physics, nuclear and particle.)
NASA TN D-1221 National Aeronautics and Space Administration. INFLUENCE OF CERTAIN COMPOSITION AND FABRICATION VARIABLES ON THE STRESS-RUPTURE PROPERTIES ON THE STRESS-RUPTURE BY POWDER METALLURGY. Philip A. Clarkin, John W. Weeton, and Paul F. Sikora. August 1962. 86p. OTS price, \$2.25. (NASA TECHNICAL NOTE D-1221) An investigation was conducted to determine the effect of carbon content and type and distribution of carbide on the stress-rupture properties and on the forgeability of an S-816 powder-metallurgy alloy. Liquid-state-sintering techniques, as well as heat treatments and mechanical working, were used to control the carbide formations and distributions. The majority of specimens produced had stress-rupture lives longer than that of standard wrought S-816 under the same test conditions. Highly forgeable structures were produced in specimens containing both high and low carbon contents. Stress-rupture lives increased with increasing carbon content.	NASA TN D-1221 National Aeronautics and Space Administration. INFLUENCE OF CERTAIN COMPOSITION AND FABRICATION VARIABLES ON THE STRESS- RUPTURE PROPERTIES ON THE STRESS- RULOY CONSOLIDATED BY POWDER METALLURGY. Philip A. Clarkin, John W. Weeton, and Paul F. Sikora. August 1962. 86p. OTS price, \$2.25. (NASA TECHNICAL NOTE D-1221) An investigation was conducted to determine the effect of carbon content and type and distribution of carbide on the stress-rupture properties and on the forgeability of an S-816 powder-metallurgy alloy. Liquid-state-sintering techniques, as well as heat treatments and mechanical working, were used to control the carbide formations and distributions. The majority of specimens produced had stress-rupture lives longer than that of standard wrought S-816 under the same test conditions. Highly forgeable structures were produced in specimens containing both high and low carbon contents. Stress-rupture lives increased
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